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US Army Research Laboratory

Verification and Demonstration for Transition of Nonhexavalent Chromium, Low–Volatile Organic Compound (VOC) Alternative Technologies to Replace DOD-P-15328 Wash Primer for Multimetal Applications

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NOTICES

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by John Kelley and Thomas Braswell
Weapons and Materials Research Directorate, ARL

Thomas Considine and Alicia Farrell
Bowhead Total Enterprise Solutions, LLC, Belcamp, MD

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14. ABSTRACT

The DOD-P-15328 wash primer is used to passivate metal surfaces to protect against corrosion and improve the adhesion of the subsequent primer to the base metal. The wash primer contains hexavalent chromium (Cr(VI)) and volatile organic compounds (VOCs) that are risks to both human health and the environment. A memo released by the Office of the Secretary of Defense in 2009 specifically directs the military to approve the use of alternatives to Cr(VI) where they can perform adequately for the intended application. This effort examines the effectiveness and demonstrates the feasibility of alternative spray-applied pretreatments for multimetal substrates as a replacement for DOD-P-15328 hexavalent chrome containing wash primer. Eight nonchromate alternative candidates were assessed against the baseline wash primer. The results show that there are viable alternatives that can provide comparable, and in some cases improved, performance to the legacy wash primer. One such candidate, a nonchromate, low-VOC product, compared very well with the baseline wash primer and is a viable drop-in replacement. Furthermore, we found that the DOD-P-15328 wash primer would not meet some of the requirements of TT-C-490F and would not be approved for use in many of the applications today. The approval of multiple candidates to populate the TT-C-490F qualified product database has prompted the US Army Research Laboratory to cancel the wash primer specification on September 30, 2017, and directs users to begin transitioning toward full implementation of TT-C-490F type III and IV pretreatments on military systems that currently rely on DOD-P-15328.

15. SUBJECT TERMS

DOD-P-15328, VOC, Cr(VI), TT-C-490F, CARC

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1. Introduction

Under the regulation AR 750-1,[1] all Army-based ground equipment is required to have a full chemical agent–resistant coating (CARC) system. The description of what typically comprises a full CARC system is defined in MIL-DTL-53072.[2] The typical CARC system consists of 1) a conversion coating or pretreatment in direct contact with the substrate properly applied in accordance with (IAW) TT-C-490,[3] followed by 2) an epoxy primer IAW MIL-DTL-530224[4] or MIL-DTL-53030[5], and 3) the polyurethane-based topcoat IAW MIL-DTL-53039[6] or MIL-DTL-64159.[7] Completed assemblies, whole platforms, and vehicles instead will require the spray-applied wash primer, DOD-P-15328,[8] as the pretreatment prior to applying the epoxy primer and polyurethane topcoat.

The DOD-P-15328 chromated wash primer was developed by the Bakelite Co. during World War II while under contract by the US government. The wash primer was developed as a substitute metal pretreatment in ship construction. It is a 2-component system consisting of a zinc chromate rust-inhibiting pigment in a flexible adhering polyvinyl butyral polymer activated by phosphoric acid prior to use. The DOD-P-15328 wash primer has been a workhorse pretreatment for the Department of Defense (DOD), performing steadfastly for many years to passivate metal surfaces to protect against corrosion and improve the adhesion of the primer to the substrate/pretreatment.[9]

It has been known for quite some time that chemical treatments containing hexavalent chromium [Cr(VI)] are risks to both human health and the environment. In April of 2009, a memo was released from Office of the Secretary of Defense that outlined a new policy for reducing the use of Cr(VI) for DoD applications. The memo specifically directs the military to approve the use of alternatives to Cr(VI) where they can perform adequately for the intended application and operating environment, update relevant technical documents and specifications to authorize the use of qualified alternatives to Cr(VI), and requires Program Executive Office or equivalent, in coordination with the Military Department's Corrosion Control and Prevention Executive, to certify that there is no acceptable alternative to the use of Cr(VI) on a new system. Effectively, the memo directs DoD military departments to restrict the use of Cr(VI) unless no cost-effective alternative with satisfactory performance can be identified.[10]

This effort examines the effectiveness and feasibility of alternative spray-applied pretreatments for multiple metal substrates to include steel, galvanized steel, stainless steel, and aluminum versus the baseline DOD-P-15328 chromated wash primer. The candidate materials were laboratory tested with the best performers

selected for full-scale trials on military assets. The alternatives that meet performance requirements of Federal specification TT-C-490F will be listed on the qualified product database (QPD) for the specification. The QPD allows applicators the option to use these nonchromate alternatives without requiring drawing changes, effectively expediting the implementation of the alternative products.

2. Experimental Procedure

2.1 Sample Preparation

Standard test coupons of 3 main sizes were used for testing the alternatives: 4 × 6 × 3/16 inch, and 4 × 12 × 1/8 inch. These standard flat test coupons were made from 4 alloys: aluminum 6061, ASTM A1008[11] cold-rolled steel (CRS), galvanized steel, and SAE 304 stainless steel.[12] In addition to these standard test coupons, a special galvanic multimetal test panel was fabricated using the 4 × 12 × 3/16 inch CRS U-weld panels as a base. These panels have a steel "U" channel welded in the center of the bottom quarter of the CRS panel and are abrasive blasted with 60-grit aluminum oxide to a surface profile of 1.5 mil. The "U" channel is positioned with the concave side facing upward when exposed. An aluminum 6061 "L" bracket was fastened to the top quarter of the CRS panel using one stainless steel screw and washer in addition to one galvanized steel screw and washer. An example of a multimetal test panel can be seen in Fig. 1. Only the steel of the galvanic multimetal panels was abrasive blasted; the standard test coupons retained a mill finish.

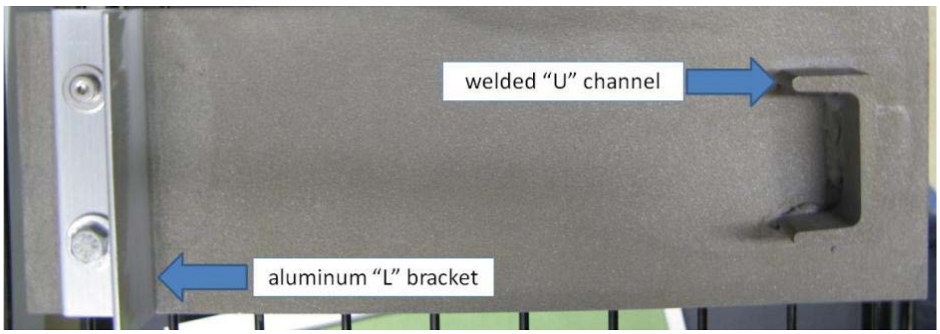


Fig. 1 Multimetal galvanic test specimen prior to pretreatment and paint

The test matrix for the project is displayed in Table 1. What is not reflected in the matrix is the preliminary screening tests performed to determine that pretreatment application parameters were within the standard wash primer. A primary objective of this study is to identify a replacement for chromated wash primer that does not significantly add to the overall processing time. The standard DOD-P-15328 wash primer used as the baseline in this program was manufactured by Sherwin Williams Company.

Table 1 Project test matrix

			Wet and Dry Cross Hatch Adhesion				Pencil Hardness ASTM D3363				SAE J400 Gravelometer				GM9540P		Outdoor Weathering		ASTM B117		Humidity Chamber	ASTM B117	GM 9540P	Outdoor Weathering
			4"x12"x1/8"				4"x12"x1/8"				4"x6"x3/16"				4x6x3/16"		4x6x3/16"		4x6x3/16"		Special multi-metal galvanic test panel			
Pretreatment	Primer	Topcoat	CRS 1008	SS 304	GAL	Al 6061	CRS 1008	SS 304	GAL	Al 6061	CRS 1008	SS 304	GAL	Al 6061	6061	CRS 1008	6061	CRS 1008	6061	CRS 1008				
Picklex 20	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022 T2	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
SurTec 650	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
AC-10	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
PPG 11-TGL-07-Z	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
Oxsilan 9810/2	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
Bonderite 7400	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
ECO 5-1	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
NCP non-chromate Wash Primer	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
DOD-P-15328 Standard Wash Primer	53030	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	53022	53039	1	1	1	1	1	1	1	1	1	1	1	1	5	5	5	5	5	5		5	5	5
	none	none																			3			
No Pretreatment	53030	53039																				5	5	5
	53022	53039																				5	5	5
	none	none																			3			

DOD-P-15328 application criteria is as follows:

- Mix ratio: 4 parts Component A and 1 part Component B
- Thin admixed material with 4 parts isopropyl alcohol
- Application is 0.3–0.5 dry mil
- Epoxy primer applied after complete drying within 30–60 min

Using the manufacturers' recommended parameters, steel test coupons were sprayed to ensure that preparation and pretreatment time prior to painting were similar to the standard wash primer. Those pretreatments that passed the initial screening were included in the test matrix.

Table 2 contains a brief description of the pretreatments examined as part of the test matrix. The list of constituents, approximate concentrations, and mix ratios* were calculated using the manufacturers' product safety data sheets and technical bulletins. Some information was not available or was listed as proprietary and is not appropriate for public release.

* It is important to note the relationship between the working solution and the mix ratio. The working solution of many of the alternatives has very few active ingredients, is water based, and contains very few VOCs.

Table 2 Candidate names and technology description

MFG/Vendor	Pretreatment	Constituents	Concentrate Approximate of Constituent (%)	Water to Concentrate Mix Ratio	Working Solution		
					Approximate of constituent (%)	Volatile Organic Compounds (%)	pH
Baseline	Chromate Wash Primer DOD-P-15328	Polyvinyl-butyral resin	N/A	N/A	6.0	85	N/A
		zinc chromate			1.7		
		magnesium silicate			0.3		
		butyl alcohol			18		
		isopropyl alcohol			67.3		
		phosphoric acid,			2.0		
NCP	Non chromate washprimer	t-butyl acetate	N/A	N/A	35-40	29	N/A
		Titanium dioxide			10-15		
		1-butanol			8		
		isobutyl alcohol			1-5		
		nitrobenzenecarboxylate			1-5		
SurTec Int	Surtec 650	trivalent chromium salts	propietary	10:1	propietary	0	3.7-3.9
		potassium flouorozirconate	<1		<0.1		
Aero-Green	AC10	Phosphoric Acid,	<10	10:1	<1.0	0	2
Henkel	Bonderite NT 7400	Flouride compound Proprietary	5-10	20:1	0.25-0.5	5	2
		1-Propoxy-2-propanol	5-10		0.25-0.5		
		Manganese compounds Proprietary	1-5		0.05-0.25		
		Hexafluorotitanic acid	1-5		0.05-0.25		
		Phosphoric acid	1-5		0.05-0.25		
Chemetall	Oxsilan 9810/2	Acidic nitrate mixture	2.5-25	25:1	0.1-1.0	1.8	4-5
		Zirconate	N/A		N/A		
		Organosilane	N/A		N/A		
International Chemical Products, Inc.	Picklex 20	Phosphoric Acid	≤7	N/A	≤7	< 1	<2
		Propietary ingredients	<8		<8		
		Water	>85		>85		
Ecosil	Ecosil 5-1	Modified Organosilanols	<40	4:1	<10	0	5
		Hexafluorozirconic acid	<1		<0.25		
PPG	PPG 11-TGL-07-Z	Hexafluorozirconic acid	N/A	N/A	0.5	0	4.6-4.8
		Propietary ingredients			propietary		

Note: All of the concentrations were provided on the safety data sheets, or by the manufacturers directly, with the exception of the DOD-P-15328 concentrations, which were obtained from specification. The NCP nonchromate wash primer concentrations were only available in weight-percent. All others are in volume-percent.

Pretreatments were applied IAW each manufacturer's process by US Army Research Laboratory (ARL) and Aberdeen Test Center (ATC) personnel. In some cases, a representative from the pretreatment manufacturer was on hand to observe the application process. All pretreatments were spray-applied per manufacturers' recommended procedures. The pretreatments were given the manufacturers' recommended time to cure, after which the test coupons were painted by ATC personnel at the ATC paint shop under ARL supervision (Fig. 2). Test coupons were primed with either MIL-DTL-53030 Type IV or MIL-DTL-53022 Type II and top coated with MIL-DTL-53039 Type II. ARL recorded both dry and wet film thicknesses (Table 3).

Fig. 2 Pretreatment application (left) and coating application at ATC (right)

Table 3 Film thicknesses for full coating systems

Pretreatment	Coating System	Al	CRS	Galv	SS
Picklex 20	53030/53039	5.58	4.48	6.44	5.36
	53022/53039	5.24	6.68	5.6	7.18
SurTec 650	53030/53039	4.94	3.8	4.8	4.4
	53022/53039	5.66	5.56	5.74	6.68
Aero-Green AC-10	53030/53039	6.92	4.16	5.22	3.66
	53022/53039	5.86	5.4	4.3	6.74
PPG 11-TGL-07-Z	53030/53039	4.72	5.16	5.2	5.78
	53022/53039	6.44	6.64	5.56	8.72
Oxsilan 9810/2	53030/53039	4.44	5.88	4.78	4.28
	53022/53039	4.74	7.48	4.66	4.42
Bonderite 7400	53030/53039	3.9	5.52	4.34	5.66
	53022/53039	6.82	4.98	8.32	8.22
Ecosil Eco 5-1	53030/53039	4.14	5.32	4.52	4.48
	53022/53039	5.86	5.1	4.92	4.52
NCP WP N-8237-2.5 A/B	53030/53039	6.04	7.04	6.02	7.68
	53022/53039	8.8	8.72	6.18	8.2
DOD-P-15328 WP	53030/53039	4.26	4.4	4.64	5.68
	53022/53039	5.9	7.78	7.4	7.24

2.2 Wet Tape Adhesion

Wet tape adhesion testing evaluates the coating's ability to resist penetration by water. This test is performed IAW Method 6301 of FED-STD-141[13] and rated per ASTM D3359[14] Method A. An "X" scribe is required on all test panels. Aluminum, CRS, galvanized steel, and stainless steel coupons were evaluated in the 24-h wet tape adhesion test. The samples were immersed in distilled water for 24 h at ambient temperature. The panels were then removed from the water and dried by wiping with a soft cloth. Two parallel lines were scribed approximately 1 inch apart with an "X" scribed between the 2 lines, ensuring that the coating was scribed through

to the substrate. A complete lap of tape was removed from the roll and discarded prior to removing the length of tape used for the test. The tape was removed from the roll at a constant, steady rate and cut to a length to completely cover the scribed area. The tape was then placed over the scribes and smoothed out by rolling with a 3-lb roller. The tape selected for this testing was 3M 250 Flatback Masking Tape, as approved by the CARC Commodity Manager and TT-C-490F. The tape was removed at a rapid, constant rate at an angle of approximately 180° to the surface. The areas around the scribes were inspected for peel-away/delamination, and the unscribed, immersed area was inspected for blistering. Each panel was rated and photo-documented per ASTM D3359[14] Method A. To further eliminate potential bias, a second rating was made by a different ARL researcher. The 2 ratings were averaged together to create the final rating.

2.3 Dry Tape Adhesion

Tests were conducted at room temperature as defined in ASTM D3359[14] Method B. An area of the panel free of blemishes was selected. A sharp tool was used to make 6 parallel cuts at 2 mm apart through the paint film to the metal substrate. A second series of cuts normal to the plane of the initial set were then made. Both cuts were made, ensuring that they were sufficiently long enough to make a complete set of 6 × 6 grid lines. Ten data points per coupon were collected. The grid lines were then brushed lightly to remove any detached flakes or ribbons of coating. A complete lap of tape was removed from the roll and discarded prior to removing the length of tape used for the test. A length of tape was removed at a steady (that is, not jerked) rate and cut about 75 mm (3 inches) long. The center of the tape was placed over the grid and the area of the grid smoothed into place by a finger. To ensure good contact with the film, the tape was rubbed firmly with the eraser on the end of a pencil. The tape was then removed by seizing the free end and rapidly pulling back upon itself at as close to an angle of 180° to the surface of the panel as possible. Following the tape pull-off, each grid was rated using the classification in ASTM D 3359 Method B, as shown in Fig. 3. To further ensure accuracy, a second rating was made by a different ARL researcher. The 2 ratings were averaged to create the final rating.

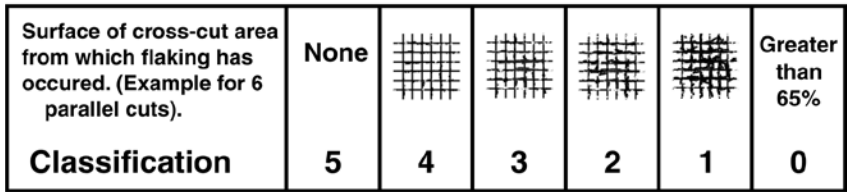


Fig. 3 ASTM D3359 Method B: crosshatch adhesion ratings

2.4 Pencil Hardness

A set of Staedtler Lumograph graphite drawing pencils was used to obtain pencil hardness values of the coating system. The coating system was allowed a full 7 days to cure in controlled laboratory conditions. Testing was done in accordance with ASTM D3363,[15] *Standard Test Method for Film Hardness by Pencil Test*. In pencil hardness testing, the pencil is first prepared using a draftsman-type mechanical pencil sharpener to expose the cylinder of lead within the pencil. The lead is then placed normal to a piece of 400-grit sand paper and ground to have a flat, chip-free surface. Starting with the hardest value pencil, in this case 4H, the operator holds the pencil at a 45° angle to the surface of the panel and pushes the pencil into the coating for 1/4 inch. If the pencil tip penetrates into the coating, the next grade of pencil trending softer is selected and the test is redone until such a point that the pencil is unable to penetrate the coating. The hardness value of the pencil that is incapable of penetrating the coating is recorded as the pencil hardness value of the coating. Figure 4 lists the pencil hardness designations. The pencil hardness test can also expose catastrophic adhesion failures with respect to the coating and the substrate. This testing was done concurrently by 2 ARL researchers, and their results averaged to eliminate potential strength biases.

4H	3H	2H	H	F	HB	B	2B	3B	4B
	←	Harder				Softer →			

Fig. 4 Pencil hardness scale

2.5 Chip Resistance Test

Prior to beginning the tests, each panel was digitally photo documented. The panels were then subjected to chip resistance testing IAW SAE J400[16] at ambient temperature using a Q-Lab Gravelometer (Fig. 5, left). The panels were held in a 45° angle specimen holder, and compressed air was used to propel gravel at the sample. The test sample was then removed and gently wiped off with a clean cloth. Tape (3M no. 898 filament strapping tape as specified in SAE J400) was then applied to the entire tested surface to remove any loose fragments of the coating.

The tested panel was then compared to standard SAE transparencies to determine a chipping rating (Fig. 5, right).

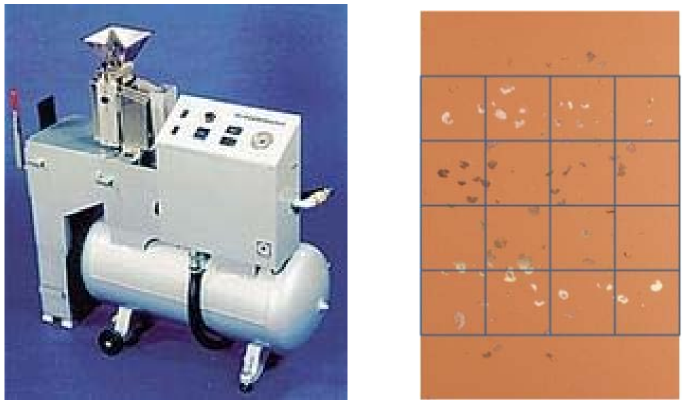


Fig. 5 Example of the Q-lab gravelometer used to measure chip resistance per SAE J400 (left) and the area of a panel evaluated (right)

Panels were again digitally photographed following tests and rated IAW SAE J400; ratings for each panel were recorded. The total number of chips inside a 4- × 4-inch grid (16 inches^2 area) using a transparency overlay was counted, and the rating obtained using Table 4. The average size of the chips was measured and rated using Table 5. For panels without a dominant chip size, the second most prevalent chip size was included (e.g., a "B/A" rating had at least 2/3 chips of size "B" and 1/3 chips of size "A").

Table 4 SAE J400 ratings for number of chips in a 4- × 4-inch area

SAE J400 Rating	10	9	8	7	6	5	4	3	2	1	0
Number of Chipps in 4"x 4" Grid	0	1	2-4	5-9	10-24	25-49	50-74	75-99	100-149	150-250	>250

Table 5 SAE J400 ratings for size of chips in a 4- × 4-inch area

A	< 1mm	(approximately < 0.03")
B	1-3mm	(approximately 0.03-0.12")
C	3-6mm	(approximately 0.12-0.25")
D	> 6mm	(approximately > 0.25")

2.6 Accelerated Corrosion Testing

Two accelerated corrosion test chambers were used to evaluate the steel test panels: the Harshaw Model 22 for standard ASTM B117[17] and an Atotech Model CCT-NC-30 for cyclic corrosion using GM9540P.[18] Figure 6 shows both test chambers in the laboratory where the testing was carried out. Test panels in each corrosion test were "X" scribed, except for the multimetal panels, which had only a single diagonal scribe that was dictated by panel geometry. In each case, the test panels were scribed through the coating to the substrate. The samples were then placed in their respective chambers, leaning at an angle between 15° and 30° from the vertical with the scribed surface facing upwards.

Fig. 6 The chamber used to conduct the neutral salt fog ASTM B117 (left) and the chamber used to conduct GM9540P tests (right)

The ASTM B117 neutral salt fog conditions are 95 °F with saturated humidity and atomized fog of 5% sodium chloride (NaCl) solution. The GM 9540P test consists of the 18 separate stages that are listed in Table 6 and includes the following intervals: saltwater spray, humidity, drying, ambient, and heated drying. The standard 0.9% NaCl, 0.1% CaCl2, 0.25% NaHCO3 test solution was used. The cyclic chamber was verified with standard steel mass loss coupons as described in the GM 9540P test specification. The salt fog chambers were verified using temperature, pH, and deposition rate measurements as per ASTM B117.

Table 6 Cycle details for the GM 9540P cyclic corrosion test

Interval	Description	Time (min)	Temperature (±3 °C)
1	Ramp to salt mist	15	25
2	Salt mist cycle	1	25
3	Dry cycle	15	30
4	Ramp to salt mist	70	25
5	Salt mist cycle	1	25
6	Dry cycle	15	30
7	Ramp to salt mist	70	25
8	Salt mist cycle	1	25
9	Dry cycle	15	30
10	Ramp to salt mist	70	25
11	Salt mist cycle	1	25
12	Dry cycle	15	30
13	Ramp to humidity	15	49
14	Humidity cycle	480	49
15	Ramp to dry	15	60
16	Dry cycle	480	60
17	Ramp to ambient	15	25
18	Ambient cycle	480	25

2.7 Outdoor Exposure Testing at Cape Canaveral Air Force Station (CCAFS)

CRS, Al 6061, and multimetal specimens were shipped from ARL to CCAFS for outdoor testing at the ARL beach exposure site. Cape Canaveral is considered one of the most corrosive environments in the continental United States. The corrosion rate observed by ASTM is 5.17 mil per year (mpy) on standard steel mass loss coupons at 55 m inland.[19] For this reason, ARL has selected this outdoor exposure facility for much of its outdoor testing. The ARL corrosion racks are set at approximately 170 m inland and parallel to the ocean facing southeast (Fig. 7). The average corrosion rate in mpy observed by ARL since 2011 on standard mass loss coupons is 1.82 mpy at 170 m inland (see Appendix A). The test panels are scribed IAW ASTM D1654 and held in place on wood composite racks with nylon standoffs and stainless steel fasteners (Fig. 8). The coupons are inspected and evaluated biannually in March and September in accordance with ASTM D1654 for both corrosion creep from the scribe as well as blistering in the field, as indicated by TT-C-490F.

Weather data (see Appendix B) are collected using a data-logging weather station and downloaded annually. As stated previously, the average rate of corrosion shown in Appendix A, since 2011, has been approximately 1.8 mils/yr. This observed corrosion rate is reasonably close to the target rate for mass loss coupons in GM 9540P of approximately 1.4 mils/yr. According to Weatherbase,[20] over the past 28 years the average temperature has been 71 °F and average rainfall is

44.8 inches. The observed average temperature during the exposure period was 74.8 °F and the average observed rainfall was 25.3 inches.

Fig. 7 Satellite image of CCAFS/TACOM outdoor exposure site in relation to ocean

Fig. 8 Example of racks on CCAFS/TACOM outdoor exposure site

2.8 Hydrogen Embrittlement Testing

Hydrogen embrittlement was assessed IAW ASTM F519,[21] using Type 1d steel-notched C-ring specimen. The objective of this test is to determine if the wash primer alternatives induced hydrogen embrittlement. Four C-ring test specimens for each wash primer alternative were preloaded to 65% of the notched fracture strength of the steel and pretreated by ARL prior to testing. Each specimen was inspected for cracks after 150 h using 25X optical microscope. The hydrogen embrittlement testing was conducted for ARL by ATC. Report 2013-MM-032 in Appendix C describes the test procedure in more detail.

2.9 ASTM D4541 Pull-off Adhesion

Pull-off adhesion was performed using the PosiTest AT-A adhesion tester. This unit is a digitally controlled hydraulic unit and applies continuous pressure to the dolly adhered to the test surface. It includes a data collector that obtains the pull rate, pressure at failure, and duration of pull. ARL used 20-mm dollies in this testing. Failure modes were observed, recorded, and photographed. Tests were limited to 4 pulls per trailer to minimize the affected area. These readings were averaged together for the final report. Touch-up was performed by Letterkenny Army Depot (LEAD) personnel using a brush-on technique of MIL-DTL-53022.

2.10 Film Thickness Measurements

Film thickness was determined in accordance with ASTM D4138. A Tooke Gauge with a 2x cutting tip was used to determine the film thicknesses of the primers and topcoats on all of the coated demonstration assets. A mark was made on the surface of the topcoat using a black marker, and a groove-cutting instrument cut through all layers of coatings where the mark had been made and down to the substrate. A microscope with measuring reticle was then used to determine the depth of each layer of coating. Additionally, a camera was affixed to the microscope to photo-document the measurements.

2.11 Performance Objectives

The performance objectives and the success criteria are defined in Table 7. The success criteria for all of the pretreatments tested were based on several factors. The first source considered was TT-C-490 revision F. For products to be qualified for use, they must meet the requirements for inclusion in the specification QPD. These requirements have been integrated into the performance objectives in Table 7. Additional requirements that are not reflected in TT-C-490F were determined using the relative performance of the alternatives to the standard DOD-P-15328 wash primer.

Table 7 Performance objectives with success criteria

Performance Objective	Data Requirements	Success Criteria
Adhesion Test	ASTM D3359 Dry Adhesion ASTM- D3359 Wet Adhesion/Fed Std 141 6301.3	Adhesion rating (all substratesl) ≥ 4; Adhesion rating (all substratesl) ≥ 4;
Coating Hardness	Pencil Hardness ASTM D3363	Film softening shall not exceed two pencil hardness difference from baseline
Chip Resistance	SAE-J400	All substrates: Rating for number of chips ≥ 5 Rating for size of chips ≥ B
Corrosion Resistance	ASTM B117 Salt Fog ASTM D1654	After 336/1000* hours of exposure: Steel substrate rating ≥ 6 scribed,≥ 7 BIF** Aluminum substrate rating ≥ 8 scribed, ≥ 7 BIF** Multimetal combined rating ≥ 6 scribed,≥ 7 BIF**
	GM 9540P Cyclic Corrosion ASTM D1654	After 40 cycles of exposure***: Steel substrate rating ≥ 6 scribed, ≥ 7 BIF** Aluminum substrate rating ≥ 8 scribed, ≥ 7 BIF** Multimetal combined rating ≥ 6 scribed, ≥ 7 BIF**
Flash Rust inhibition	modified ASTM D1735	No flash rust after 24 hours of exposure to ambient temperature and 90% relative humidity
Outdoor Exposure	Cape Canaveral ASTM D1654 ASTM G50	Two years of exposure: Steel substrate rating ≥ baseline Aluminum substrate rating ≥ baseline Multimetal combined ratings ≥ baseline
Hydrogen Embrittlement	ASTM F519	No cracking after 150 hours
Toxicity Clearance	Toxicity clearances and full disclosure from Public Health Command	Approved by processing facility
Processing time	TT-C-490F	Equivalent or less than existing process
Field Testing	TT-C-490F	After 2 year outdoor exposure rating ≥ current technology
Qualitative Performance Objectives		
Ease of use	Feedback from field technician on usability of technology and time required during demonstration	No formal operator training required. Training from supplier technical representative.

* 336 hours for MIL-DTL-53022 Type II, 1000 hours for MIL-DTL-53030 Type IV
** Blisters in Field (BIF), no single blister in excess of 3mm
*** No cyclic requirement for MIL-DTL-53022 Type II

3. Results and Discussion

3.1 Lab Results

3.1.1 Wet Tape Adhesion

All adhesion testing was done on mill finish panels. That is, there was no abrasive blasting or appreciable profile on any of the 4 substrates tested (aluminum, cold rolled steel, galvanized steel, and stainless steel). Table 8 lists the ratings for the wet tape adhesion tests for all candidates and baselines along with the color coding for met/not met of the success criteria listed in Table 7 for wet tape adhesion (green = met and red = not met). The success criterion for wet tape adhesion is a rating greater than or equal to 4. The baseline DOD-P-15328 wash primer performed well, meeting the success criterion with both MIL-DTL-53022 and MIL-DTL-53030 primers on all substrates. The Oxsilan 9810/2 and Ecosil (ECO 5-1) performed better than the wash primer, achieving ratings of 5 in many cases. PPG 11-TGL-07-Z (Zircobond), Bonderite M-NT-7400, and SurTec 650 showed similar adhesive performance as the wash primer and met the success criterion in most cases, galvanized surfaces being the exception. The SurTec 650 clearly did not perform well on galvanized surfaces, while the Bonderite M-NT-7400 failed with only the MIL-DTL-53030 primer.

Table 8 Wet tape adhesion ratings (ASTM D3359 method A)

		Wet Adhesion			
		Al	CRS	Galv	SS
Picklex 20	53022	0	2	3	0
	53030	3	3	3	4
SurTec 650	53022	4	4	2	4
	53030	4	5	2	5
Aero-Green AC-10	53022	3	4	5	0
	53030	4	3	3	4
PPG 11-TGL-07-Z	53022	4	4	4	4
	53030	4	4	5	4
Oxsilan 9810/2	53022	4	4	4	4
	53030	5	5	5	4
Bonderite 7400	53022	4	5	5	4
	53030	5	5	0	5
Ecosil Eco 5-1	53022	5	5	5	5
	53030	5	5	5	4
NCP WP N-8237-2.5 A/B	53022	0	1	0	1
	53030	0	3	3	2
DOD-P-15328 WP	53022	4	4	4	4
	53030	4	4	4	4

Picklex, AC-10, and the NCP nonchromated wash primer did not meet the minimum requirements for the wet tape adhesion criterion across most substrate/primer combinations. On average, Ecosil outperformed the wash primer and all other candidates in wet tape adhesion with all substrate/primer combinations.

3.1.2 Dry Tape Adhesion

As in the wet tape adhesion testing, dry tape adhesion (or crosshatch) testing was done on mill finish versions on the same 4 substrates as above. No appreciable surface profile was given to any of the substrates.

Table 9 lists the ratings for the dry tape adhesion tests for all candidates and baselines along with the color coding for met/not met success criteria listed in Table 7 for dry tape adhesion (green = met and red = not met). The success criterion for dry tape adhesion is a rating greater than or equal to 4. As with the wet tape adhesion, the baseline DOD-P-15328 wash primer met the success criterion with both MIL-DTL-53022 and MIL-DTL-53030 primers on all substrates.

Table 9 Dry tape adhesion ratings (ASTM D3359 method B)

		Cross Hatch Adhesion			
		Al	CRS	Galv	SS
Picklex 20	53022	0	0	4	4
	53030	0	1	4	4
SurTec 650	53022	2	4	0	4
	53030	4	3	2	5
Aero-Green AC-10	53022	3	3	5	0
	53030	3	2	2	4
PPG 11-TGL-07-Z	53022	4	3	5	4
	53030	4	4	3	5
Oxsilan 9810/2	53022	5	4	5	5
	53030	5	3	5	4
Bonderite 7400	53022	4	4	4	4
	53030	5	5	2	5
Ecosil Eco 5-1	53022	5	5	5	5
	53030	5	5	4	5
NCP WP N-8237-2.5 A/B	53022	0	0	0	2
	53030	0	4	4	4
DOD-P-15328 WP	53022	4	4	4	4
	53030	4	4	4	4

A similar trend is seen in dry tape adhesion testing as was seen in wet tape adhesion testing regarding how each product performs on each substrate with each primer. Oxsilan 9810/2, Ecosil, and Bonderite M-NT-7400 performed similar to or better than the wash primer in most situations. PPG 11-TGL-07-Z (Zircobond) and SurTec 650 performed less favorably than they had in wet adhesion testing. Overall, the SurTec 650, Picklex, AC-10, and NCP nonchromated wash primer performed at a substantially lower level than the baseline wash primer. The difference in performance of the primers was much less distinct in dry adhesion testing. There was also not much difference in the adhesive performance of the wash primer alternatives across each substrate, save for stainless steel, which was almost an entire rating point higher than the other 3 substrates when averaged. Overall, Ecosil again outperformed the wash primer and all other candidates in dry tape adhesion with all substrate/primer combinations.

3.1.3 Pencil Hardness

There was little variation in the pencil hardness of the coatings across each substrate/primer/pretreatment combination. The success criteria in Table 7 state that film softening shall not exceed a 2-pencil-hardness difference from the baseline. The DOD-P-15328 has a baseline pencil hardness rating for each primer. For MIL-DTL-53022, the baseline hardness is “B”, and for MIL-DTL-53030 the baseline hardness is HB. All of the combinations in Table 10 met the success criteria for pencil hardness, indicating that the pretreatments had no significant effect on the hardness of the coating system. The hardest rating was an “F” and was on aluminum and stainless steel panels treated with Oxsilan and primed with MIL-DTL-53022. The lowest reported pencil hardness was 2B and was seen on many of the NCP wash primer combinations, but was still within the 2-hardness rating requirement. The 2B grades seen on the NCP wash primer reflect the issues with adhesion displayed in the previous 2 adhesion tests.

Table 10 Pencil hardness ratings (ASTM D3363)

		Pencil Hardness			
		Al	CRS	Galv	SS
Picklex 20	53022	HB	B	B	B
	53030	HB	HB	B	HB
SurTec 650	53022	HB	HB	HB	HB
	53030	B	HB	HB	2B
Aero-Green AC-10	53022	2B	B	B	B
	53030	B	B	B	B
PPG 11-TGL-07-Z	53022	HB	HB	HB	B
	53030	HB	HB	B	HB
Oxsilan 9810/2	53022	F	2B	HB	F
	53030	HB	B	B	HB
Bonderite 7400	53022	HB	HB	B	HB
	53030	B	B	B	HB
Ecosil Eco 5-1	53022	HB	HB	HB	B
	53030	B	B	HB	HB
NCP WP N-8237-2.5 A/B	53022	2B	2B	HB	B
	53030	2B	2B	2B	2B
DOD-P-15328 WP	53022	B	B	B	HB
	53030	B	HB	HB	HB

3.1.4 Chip Resistance Testing

Table 11 shows the ratings for SAE J400 chip resistance. The pretreatments varied across each of the substrates, with stainless steel having the lowest ratings. The Bonderite M-NT-7400, Ecosil ECO 5-1, the baseline wash primer, Oxsilan 9810/2, and PPG 11-TGL-07-Z performed well regardless of substrate. The Picklex product showed poor resistance to chipping and was the poorest performer of all the products, unable to meet the success criterion on any substrate. The chip resistance test results appear to be congruent with the results seen in the previous 3 adhesion tests. In general, those that provided the best adhesion were also the most resistant to chipping.

Table 11 Chip resistance ratings per SAE J400

Pretreatment	Primer	Chip Resistance, SAE J400 Ratings			
		Al	CRS	Galv	SS
Picklex	53022	1D	8D	9C	5D
	53030	2D	5D	7C	5D
SurTec 650	53022	7A	9D	8D	6C
	53030	6A	6A	6A	6B
AC-10	53022	6C	9A	9A	5D
	53030	4D	8B	8C	2C
PPG 11-TGL-07-Z	53022	7B	7A	7A	7C
	53030	7B	6A	6A	6C
Oxsilan 9810/2	53022	7A	7A	8A	7B
	53030	7B	6A	6A	6C
Bonderite 7400	53022	7A	8A	6B	6C
	53030	7B	6A	7A	7B
Ecosil ECO 5-1	53022	8A	8A	9B	7B
	53030	7A	7A	7A	5C
NCP WP	53022	5C	9B	8A	7D
	53030	6D	7C	6B	5D
DOD-P-15328	53022	7C	7A	7A	5B
	53030	6B	6A	6A	5B

Figures 9–12 show the results of the SAE J400 test panels after testing. Although the ratings quantitatively determine whether the pretreatments met or did not meet the success criteria, Figs. 9–12 show the performance qualitatively. Ecosil 5-1 and the Bonderite M-NT-7400 were 2 of the most resistant to chipping along with the Oxsilan 9810/2 (not shown). These alternatives compare very well to the baseline wash primer in Fig. 11. In fact, quantitatively, one could deduce the alternatives were better at resisting chipping. In contrast, Fig. 12 shows the poor performance of the Picklex with all primers and substrates. The chip resistance is particularly important for ground vehicles to prevent damage to the CARC system.

Fig. 9 Bonderite M-NT-7400 on (from L to R) aluminum, cold-rolled, galvanized, and stainless steel (53030 top, 53022 bottom)

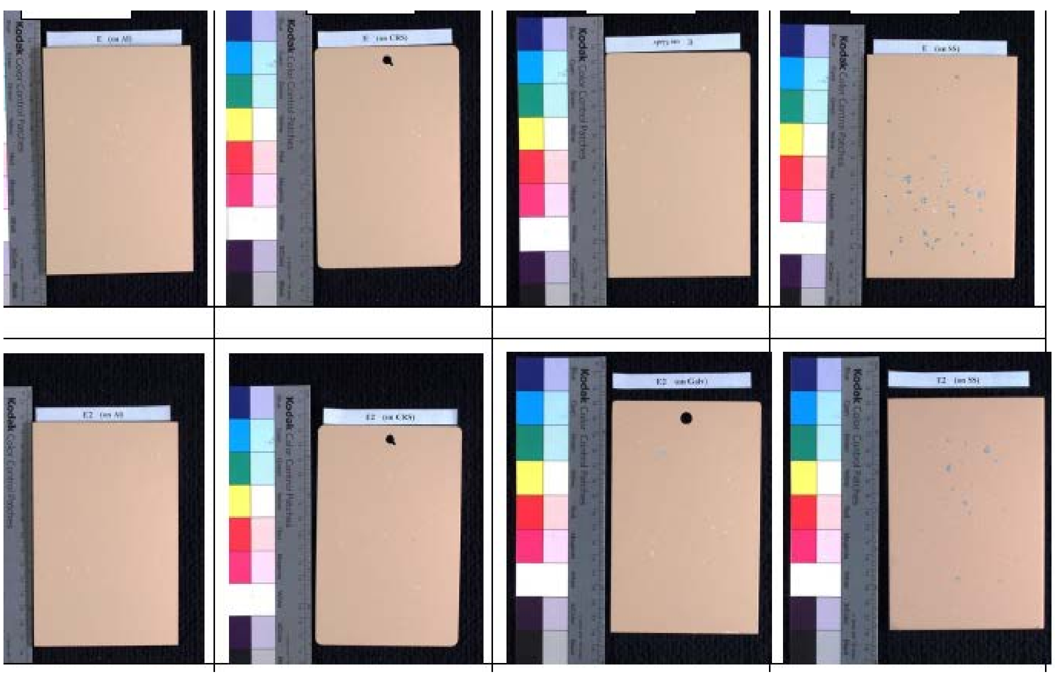


Fig. 10 Ecosil 5-1 on (from L to R) aluminum, cold-rolled, galvanized, and stainless steel (53030 top, 53022 bottom)

Fig. 11 DOD-P-15328 wash primer on (from L to R) aluminum, cold-rolled, galvanized, and stainless steel (53030 top, 53022 bottom)

Fig. 12 Picklex on (from L to R) aluminum, cold-rolled, galvanized, and stainless steel (53030 top, 53022 bottom)

3.1.5 Neutral Salt Fog Corrosion (ASTM B117)

The success criteria for testing IAW ASTM B117 salt fog was derived from TT-C-490F and is listed in Table 7. MIL-DTL-53022 and MIL-DTL-53030 primers are evaluated after 336 and 1008 h, respectively. To meet the performance objectives, steel test panels must achieve a creepage from scribe rating ≥6 and blisters in field must be ≥7. For aluminum panels, the creepage from scribe rating shall be ≥8 and blisters in field ≥7. The multimetal specimens use a rating for the aluminum section combined with the steel section and the ratings shall be ≥6 scribed, with ≥7 blisters in field. In all cases, no single blister in field shall exceed 3-mm width.

CRS panels primed with MIL-DTL-53030 and MIL-DTL-53022 ran for 1008 h in salt fog testing. The ASTM D1654 ratings for all pretreatments and baseline on CRS can be seen in Table 12. All pretreatments met the success criteria of ≥6 after 336 h with MIL-DTL-53022. When evaluated at 1008 h for the MIL-DTL-53030, the only pretreatments that met the success criteria were Bonderite M-NT-7400, baseline wash primer, and the NCP wash primer. There was no blistering in the field on any panel through 1008 h, though staining from the edges and the scribe became prevalent over time. These stains are not accounted for during the rating process. Figures 13 and 14 is a visual comparison of the Bonderite M-NT-7400 versus the baseline wash primer. The TT-C-490 requirement is a rating of ≥6 at 336 h for the 2 primers used. The 336-h results show that many of the pretreatments tested would meet the requirements for qualification on steel substrates.

Table 12 ASTM D1654 ratings for ASTM B117 neutral salt fog testing on CRS

		Exposure Time (hours)					
		Creep from Scribe			Blisters in Field		
Pretreatment		336	672	1008	336	672	1008
Picklex 20	53022	7.6	6.4	5	10	10	10
	53030	4.4	3.6	3.6	10	10	10
SurTec 650	53022	8.2	6.6	6.4	10	10	10
	53030	8	5.8	5.6	10	10	10
Aero-Green AC-10	53022	6.6	5.6	5	10	10	10
	53030	6	5.6	4	10	10	10
PPG 11-TGL-07-Z	53022	8.6	4.4	3	10	10	10
	53030	5.2	3.6	3.4	10	10	10
Oxsilan 9810/2	53022	7.6	5.6	4.8	10	10	10
	53030	6.2	5.4	4.2	10	10	10
Bonderite 7400	53022	8.4	6.6	5.6	10	10	10
	53030	8.4	7	6	10	10	10
Ecosil Eco 5-1	53022	7.8	6.4	6	10	10	10
	53030	8	6.2	5.4	10	10	10
NCP WP N-8237-2.5 A/B	53022	7.6	7.2	7.2	10	10	10
	53030	7	6.2	6.2	10	10	10
DOD-P-15328 WP	53022	9	7.2	7	10	10	10
	53030	8.4	6.6	6.2	10	10	10

Fig. 13 Bonderite M-NT-7400 rated a 6 on steel panels with MIL-DTL-53030 at 1008 h

Fig. 14 DOD-P-15328 rated a 6.2 on steel panels with MIL-DTL-53030 at 1008 h

Aluminum panels were run out to 1008 h of exposure in ASTM B117 testing. The results obtained can be seen in Table 13. At 1008 h, only the Bonderite M-NT-7400 and DOD-P-15328 wash primer met the success criteria of a ≥8, and that is only with the MIL-DTL-53030 CARC primer. No pretreatments on aluminum with MIL-DTL-53022 met the success criteria for creep from scribe after 336 h. The Picklex-treated panels primed with 53022 had completely delaminated after 72 h, and the AC-10 treated panels primed with 53022 were completely delaminated after 168 h. The Bonderite M-NT-7400 performed slightly better than that of the baseline

wash primer. Some were very close to the required ≥8 rating. Although the SurTec 650 (trivalent chrome aluminum pretreatment) did not perform as well on steel substrates, it had consistent ratings with both primer types. Bonderite M-NT-7400 and NCP nonchromate wash primer rated a 7.4 and 7.6, respectively. The ratings for all aluminum samples are expected to be lower once the required 1008 h of salt fog exposure is reached. The reason for the low ratings on aluminum is likely because aluminum was not sufficiently cleaned and should be deoxidized prior to pretreatment. The native oxide of the aluminum hinders proper reaction of the conversion coatings. TT-C-490 requires a test length of 336 h for the 2 primers used in this study. Looking at the 336-h results, only the Bonderite M-NT 7400, SurTec 650, and chromated wash primer would meet the requirements for qualification in this case.

Table 13 ASTM D1654 ratings for ASTM B117 neutral salt fog testing on aluminum

		Exposure Time (hours)					
		Creep from Scribe			Blisters in Field		
Pretreatment		336	672	1008	336	672	1008
Picklex 20	53022	0	0	0	0	0	0
	53030	4.6	2.8	1	10	10	10
SurTec 650	53022	7.8	7.8	7.6	10	10	10
	53030	8.4	7.8	6.8	10	10	10
Aero-Green AC-10	53022	0	0	0	0	0	0
	53030	3.2	2.6	1	10	10	10
PPG 11-TGL-07-Z	53022	6.4	4.6	3.4	10	10	10
	53030	6	4	3	10	10	10
Oxsilan 9810/2	53022	7	6.4	4.4	10	10	10
	53030	2.4	1.2	0.4	10	10	10
Bonderite 7400	53022	7.8	7.4	7.4	10	10	10
	53030	9	9	9	10	10	10
Ecosil Eco 5-1	53022	3.8	3.8	2.6	10	10	10
	53030	3.6	2	0.4	10	10	10
NCP WP N-8237-2.5 A/B	53022	7.6	7.6	6.6	10	10	10
	53030	6.4	6.4	5.4	10	10	10
DOD-P-15328 WP	53022	7.2	7.2	7.8	10	10	10
	53030	9	9	9	10	10	10

Figures 15 and 16 are photographs of MIL-DTL-53030 panel sets through 1008 h. At 1008 h, both the ratings and photographs show that the Bonderite M-NT-7400 compares very well with the baseline wash primer on steel and aluminum. The Picklex shown in Fig. 17 rated 0.0, and along with AC-10, showed early catastrophic failures starting after 72 h.

Fig. 15 Bonderite M-NT-7400 rated a 9 on Al panels with MIL-DTL-53030 at 1000 h

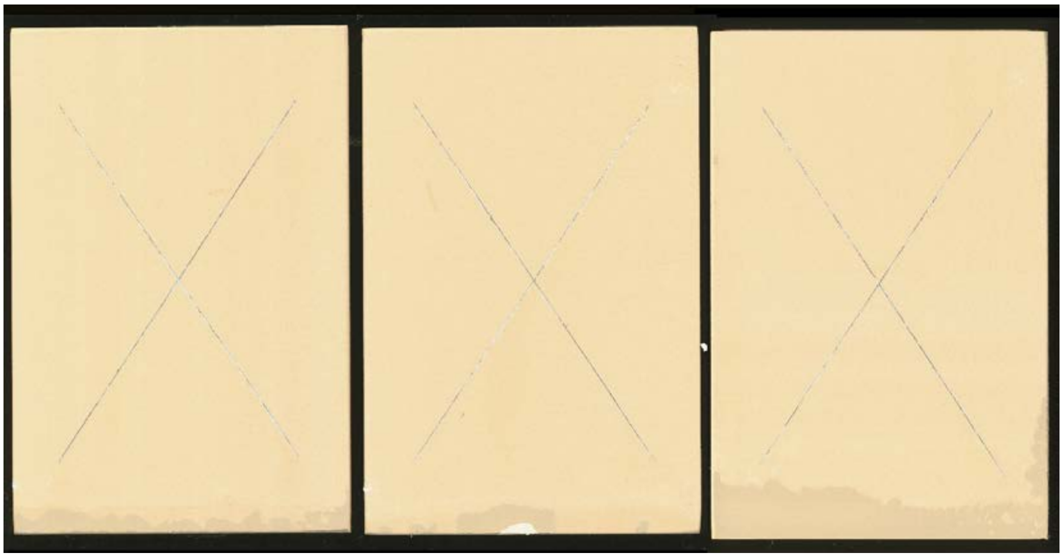


Fig. 16 DOD-P-15328 rated a 9 on Al panels with MIL-DTL-53030 at 1000 h

Fig. 17 Picklex rated a 0.0 on Al panels with MIL-DTL-53022 at only 72 h

Multimetal panels were exposed through 1008 h of B117 testing. The success criteria for these samples take into consideration all of the exposed surface area for blisters in the field. The ratings for the multimetal specimens can be seen in Table 14. Similar to what was discussed earlier with proper cleaning and deoxidizing Al, coating adhesion on the extruded aluminum "L" bracket was challenging. Additionally, the galvanized fastener and surrounding area tended to show more corrosion than that of the stainless steel fastener. The corrosion product exuded by the galvanized fastener tended to build up over time on the aluminum L bracket and was not removed by the fog condensing on the substrate or rinsing during inspections. Staining around the U-weld was common on all specimens. As can be seen in Table 14, most of the pretreatments and baseline met the success criteria, including direct-to-metal (DTM), which has no pretreatment. Interestingly, the DTM samples performed very similarly to the wash primer samples. The Bonderite M-NT-7400 performed consistently across all substrates and primers easily meeting all of the success criteria.

Table 14 ASTM D1654 ratings for ASTM B117 salt fog testing on multimetal panels

		Exposure Time (hours)					
		Creep from Scribe			Blisters in Field		
Pretreatment		336	672	1008	336	672	1008
Picklex 20	53022	6.8	5.4	3.8	7	7	5.2
	53030	8.6	8.6	6.4	9.4	7	6.6
SurTec 650	53022	7	6.2	5.8	7.6	7.2	6.4
	53030	8.2	7.2	6.8	7.8	6.2	6
Aero-Green AC-10	53022	8.2	7.6	7	7	7	6
	53030	9	7.2	5.4	9.4	8.8	7.4
PPG 11-TGL-07-Z	53022	7.2	6	5.8	9	7	6.8
	53030	7.2	6.6	5.6	8.8	8	6.8
Oxsilan 9810/2	53022	7.4	6.2	5.8	8	6.6	6.4
	53030	7.4	7.4	6.2	8.4	7.4	7.2
Bonderite 7400	53022	9	8.2	7.6	9.4	8	6.6
	53030	9	8.8	7.8	8.6	8.6	8.2
Ecosil Eco 5-1	53022	7.8	6.8	6	8	7.6	7
	53030	8.6	7	5.4	7.2	6.8	6.2
NCP WP N-8237-2.5 A/B	53022	5.4	4.8	4.6	8.6	6.6	5
	53030	8.4	7.8	6	10	9.4	8
DOD-P-15328 WP	53022	7	6.4	7	9.8	9.6	8.8
	53030	8.8	7.8	6	10	10	9
Direct to Metal	53022	9	7.4	5.4	8	6.8	5.6
	53030	7	6.4	5.6	7.6	6	5.2

An example of the performance of Bonderite M-NT-7400 versus the baseline wash primer can be seen in Figs. 18 and 19. Although the success criteria were met after 336 h of ASTM B117 exposure, these photographs were taken after 1008 h. Despite the Bonderite M-NT-7400 samples showing significantly more staining and edge corrosion, there is less undercutting at the scribe on average than the wash primer. The chromate in the wash primer is clearly affecting the appearance of rust staining as seen here and on the other alternatives. The coating adhesion provided by the Bonderite M-NT-7400 resists the undercutting better that the wash primer.

Fig. 18 Bonderite M-NT-7400 on multimetal panels with MIL-DTL-53022 after 1008 h

Fig. 19 DOD-P-15328 on multimetal panels with MIL-DTL-53022 after 1008 h

3.1.6 Accelerated Cyclic Corrosion (GM 9540P)

CRS panels were exposed to 80 cycles of GM 9540P cyclic corrosion testing; however, the success criteria are evaluated after 40 cycles of exposure (see Table 7). After 40 cycles, the steel substrate rating shall be ≥6 creep from scribe, and the blisters in field rating ≥7. For aluminum, the rating shall be ≥8 creep from scribe,

and ≥7 for blisters in field. The multimetal samples require a rating ≥6 scribed and a combined rating ≥7 blisters in the field to include aluminum and U-weld areas.

The ASTM D1654 ratings for all pretreatments on CRS are presented in Table 15. At 40 cycles, only the baseline wash primer, Bonderite M-NT-7400, and Ecosil 5-1 met the success criteria on panels primed with either MIL-DTL-53022 or MIL-DTL-53030. Bonderite maintained the highest average rating on CRS of the 3 pretreatments that passed. AC-10 was relatively close to meeting the requirements, with 3 of 5 panels passing with ratings of 6. The other 2 panels rated 5 and kept the average below the threshold acceptable rating of 6.

Table 15 ASTM D1654 ratings for GM9540P cyclic corrosion testing on CRS

		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
Pretreatment		20	40	60	80	20	40	60	80
Picklex 20	53022	6	4	3	2.2	10	10	10	8
	53030	6.25	5	5	4	10	10	10	10
SurTec 650	53022	6.2	2.6	1.4	0	10	10	10	8
	53030	6.6	4.2	3.4	3.4	10	10	10	10
Aero-Green AC-10	53022	7.4	5.8	5.4	4.8	10	10	10	10
	53030	7.6	5.6	5.4	5.2	10	10	10	10
PPG 11-TGL-07-Z	53022	5.8	3.2	1.8	0.4	10	10	10	10
	53030	6	3.5	3.5	2.5	10	10	10	10
Oxsilan 9810/2	53022	5.4	1.6	0.4	0.2	10	10	8	2
	53030	6.6	3.8	3	2	10	10	10	10
Bonderite 7400	53022	9	6.25	5.75	5.5	10	10	10	10
	53030	9	6.75	6.75	6	10	10	10	10
Ecosil Eco 5-1	53022	9	6.25	6	5.25	10	10	10	10
	53030	8.75	6	6	5.25	10	10	10	10
NCP WP N-8237-2.5 A/B	53022	5.6	3	2	0.8	10	10	10	10
	53030	6	3	2.4	1.4	10	8	8	8
DOD-P-15328 WP	53022	6.8	6.4	5.8	5	10	10	10	10
	53030	8.25	6.5	6.5	5.5	10	10	10	10

While all of the pretreatments met the blisters in field requirement, only the NCP nonchromate wash primer, Oxsilan 9810/2, and Picklex-treated panels showed any blistering through the entire 80 h.

After the 80 cycles were completed, all panels were scraped with a 2-inch flat blade putty knife after rating to unveil any previously unseen corrosion or delamination issues between the coating and the substrate. With the exception of a small amount of hidden corrosion on an AC-10 treated panel, there were no major revelations. Only the Bonderite M-NT-7400 met the success criteria with MIL-DTL-53030 and nearly met with MIL-DTL-53022 after 80 cycles. In addition to the Bonderite M-NT-7400, the Ecosil 5-1 also performed well and met the success criteria through 60 cycles. TT-C-490 requirements are set to 40 cycles of GM9540P. With respect to TT-C-490, only Bonderite M-NT 7400, Ecosil Eco 5-1, and the chromated wash primer passed cyclic corrosion testing.

Figures 20–25 illustrate the performance of the 2 alternatives that met the success criteria for GM9540P relative to the baseline wash primer. After the panels were exposed to 80 cycles and subsequently scraped, it is difficult to discern any difference in performance.

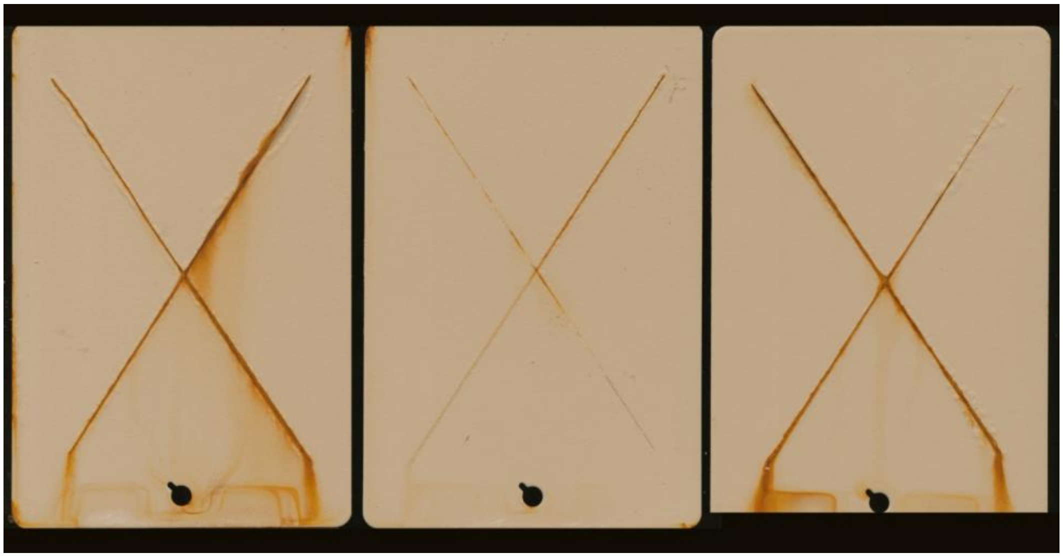


Fig. 20 Bonderite M-NT-7400 on CRS primed with MIL-DTL-53022 after 80 cycles

Fig. 21 Bonderite M-NT-7400 on CRS primed with MIL-DTL-53030 after 80 cycles

Fig. 22 Ecosil 5-1 on CRS primed with MIL-DTL-53022 after 80 cycles

Fig. 23 Ecosil 5-1 on CRS primed with MIL-DTL-53030 after 80 cycles

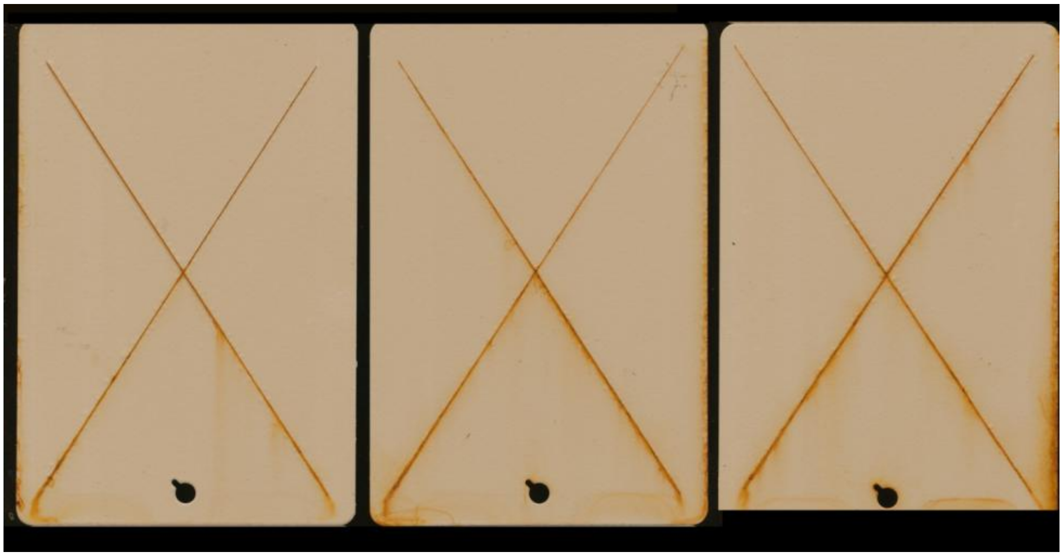


Fig. 24 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles

Fig. 25 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 80 cycles

Although success is established at 40 cycles, the aluminum test panels were also run out to 80 cycles to determine longer-term effectiveness. To meet the success criteria, the aluminum test panels must be a rated 8.0 or higher at 40 cycles. All of the ASTM D1654 ratings for GM9540P on aluminum are presented in Table 16. As discussed earlier, it is more challenging for some of these alternative pretreatments to be effective on aluminum because it is typically chemically cleaned and deoxidized as part of the pretreatment/conversion coating process. The SurTec 650 was expected to meet the success criteria here because it was developed as a conversion coating for aluminum. In addition to SurTec 650, Oxsilan 9810/2

and PPG 11-TGL-07 Z also performed well. Each of these products is a zirconium-modified pretreatment. Although the Bonderite was unable meet the success criteria with the MIL-DTL-53022 primer, it was only 0.2 below the pass threshold of an average rating of 8.

Table 16 ASTM D1654 ratings for GM9540P cyclic corrosion testing on aluminum

		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
Pretreatment		20	40	60	80	20	40	60	80
Picklex 20	53022	1.8	0	0	0	10	6	0	0
	53030	6.2	5.6	2.4	0	10	10	10	8
SurTec 650	53022	10	9	8.2	8.2	10	10	10	10
	53030	10	9	8.8	8.8	10	10	10	10
Aero-Green AC-10	53022	5	3	0	0	10	10	10	0
	53030	4.2	2.6	0.6	0	10	6	6	2
PPG 11-TGL-07-Z	53022	10	9	8	7.4	10	10	10	10
	53030	10	9	9	8.8	10	10	10	10
Oxsilan 9810/2	53022	9.6	8.6	8.6	8.4	10	10	10	10
	53030	10	9	8.2	8	10	10	10	10
Bonderite 7400	53022	8.2	7.8	7	6.8	10	10	10	10
	53030	9.8	9	8.8	8.6	10	10	10	10
Ecosil Eco 5-1	53022	9.2	8.4	6	5.8	10	10	10	10
	53030	10	9	7.2	5.2	10	10	10	10
NCP WP N-8237-2.5 A/B	53022	5.8	4	2.2	1.8	10	8	8	4
	53030	10	8.4	0	0	10	10	10	10
DOD-P-15328 WP	53022	10	8.8	8	7.8	10	10	10	10
	53030	10	9	9	8.8	10	10	10	10

It is clear that Picklex and AC-10 did not perform well on aluminum. Neither could achieve more than a 6.2 after 20 cycles. In addition, the NCP wash primer catastrophically failed, delaminating between the 40- and 60-cycle inspections with both of the primers. TT-C-490 requirements are set to 40 cycles of GM9540P. With respect to TT-C-490, only AC-10 and Picklex 20 would not meet the requirements for qualification.

The best-performing pretreatments on aluminum with either primer in cyclic testing considering success criteria were the baseline DOD-P-15328 wash primer, SurTec 650, PPG 11-TGL-07 Z Oxsilan 9810/2, Ecosil 5-1, and Bonderite M-NT-7400. Considering each product's performance on steel substrates to this point, Bonderite M-NT-7400 and Ecosil 5-1 have shown promise as viable alternatives to the baseline wash primer. Figures 26–31 show little difference in the visual appearance between the baseline wash primer, Bonderite M-NT-7400, and the Ecosil 5-1 after 80 cycles. Even with the lower creep from scribe ratings, both appear suitable for use on aluminum.

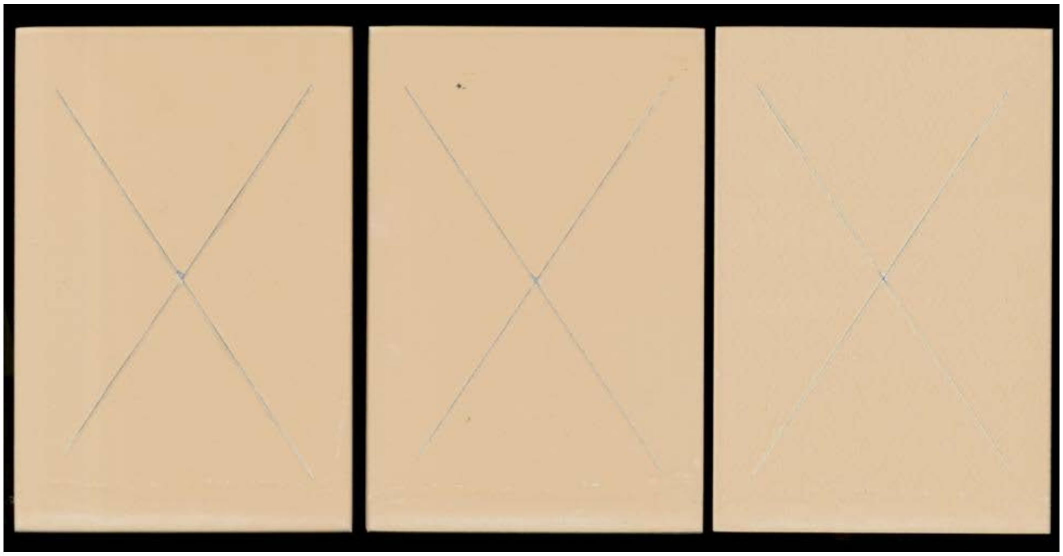


Fig. 26 Bonderite M-NT-7400 on aluminum primed with MIL-DTL-53022 after 80 cycles

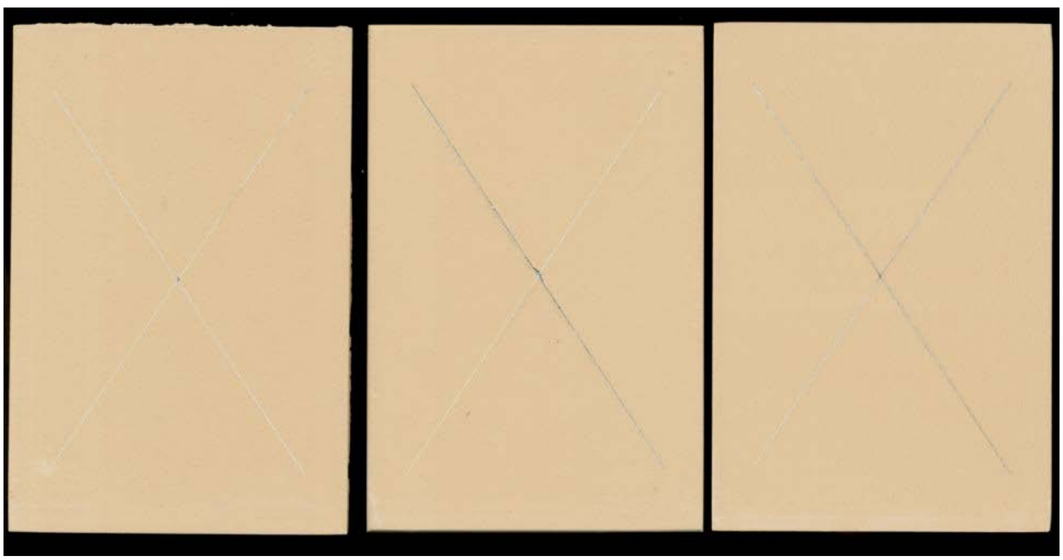


Fig. 27 Bonderite M-NT-7400 on aluminum primed with MIL-DTL-53030 after 80 cycles

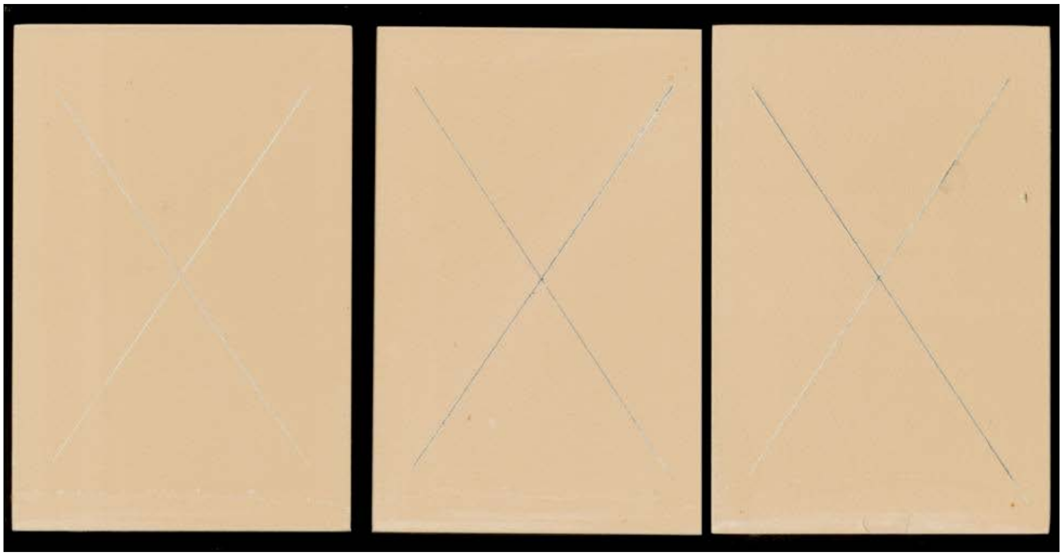


Fig. 28 Ecosil 5-1 on aluminum primed with MIL-DTL-53022 after 80 cycles

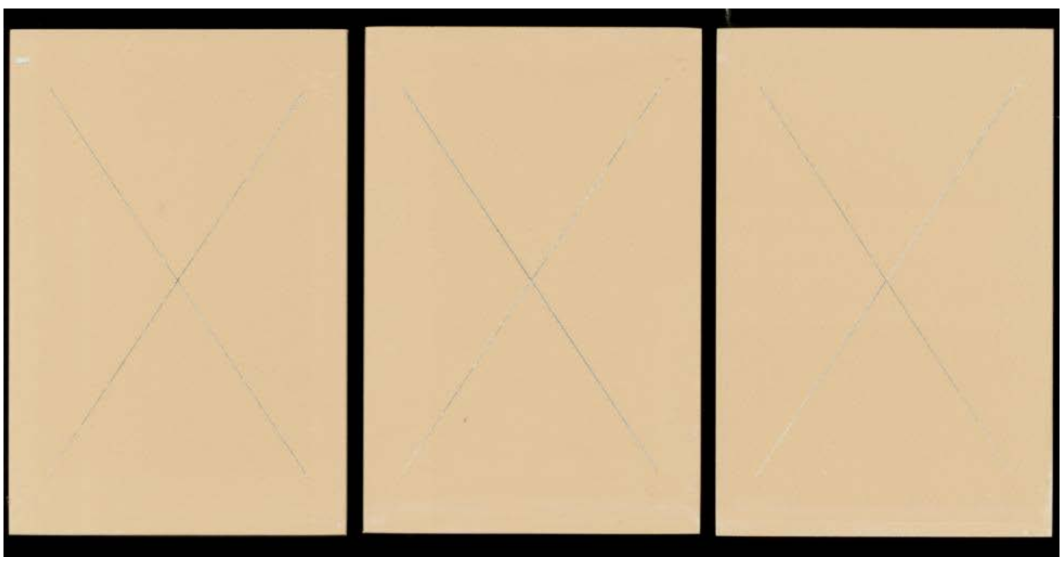


Fig. 29 Ecosil 5-1 on aluminum primed with MIL-DTL-53030 after 80 cycles

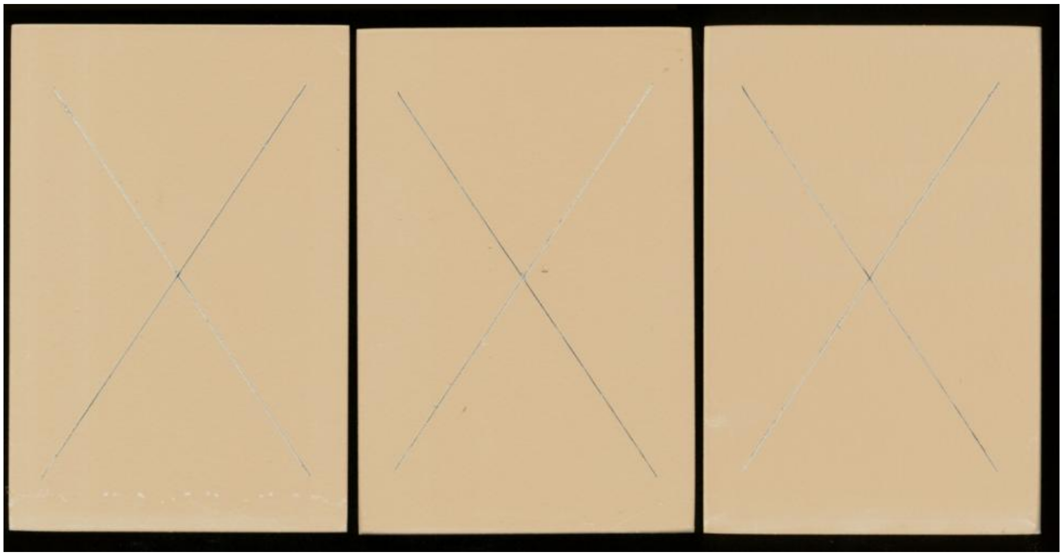


Fig. 30 DOD-P-15328 on aluminum primed with MIL-DTL-53022 after 80 cycles

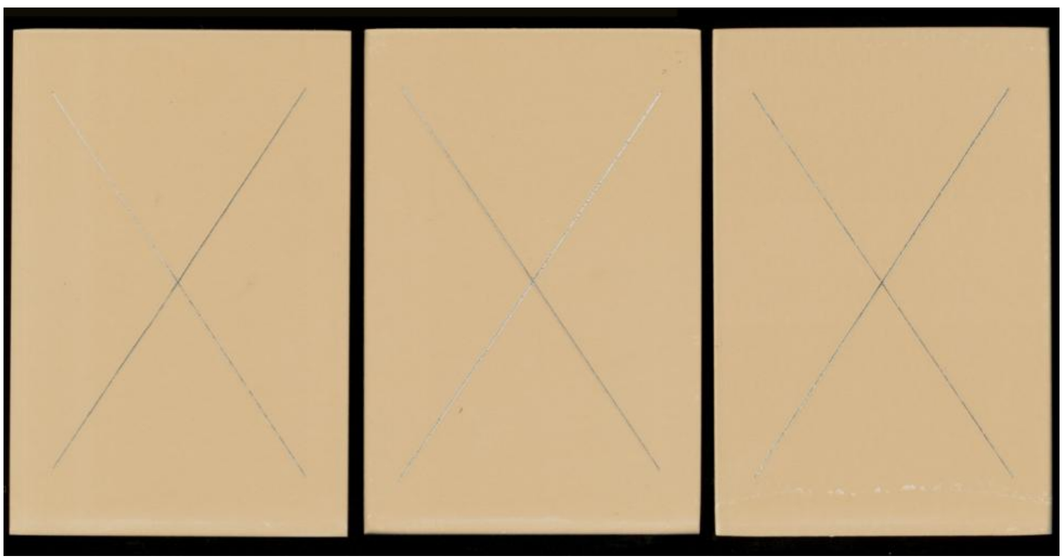


Fig. 31 DOD-P-15328 on aluminum primed with MIL-DTL-53030 after 80 cycles

Because of the more complex geometry and dissimilar metals, it was expected that this sample would be more challenging for all pretreatments including the baseline wash primer. This expectation is verified with the results presented in Table 17. The multimetal samples require a rating ≥6 scribed, and a combined rating ≥7 blisters in the field to include aluminum and U-weld areas after 40 cycles. Only 2 pretreatments were able to meet the success criteria with both primers at 40 cycles: Bonderite M-NT-7400 and Oxsilan 9810/2. In fact, the only alternatives that met the success criteria with the MIL-DTL-53022 primer were the Bonderite M-NT-7400 and the Oxsilan 9810/2. Beyond that, no pretreatment was completely

effective with both MIL-DTL-53022 and MIL-DTL-53030. Similarly, there was a significant amount of variation in ratings for blisters in the field. The Bonderite M-NT-7400 and the Oxsilan 9810/2 both met the success criteria, as did the baseline. The MIL-DTL-53030 type IV clearly provided better corrosion protection than the MIL-DTL-53022 type II in this study. Many of the alternatives performed as well as or better than the baseline wash primer when primed with the MIL-DTL-53030 primer. The only alternative pretreatment system that failed at 40 cycles with the MIL-DTL-53030 primer was the NCP nonchromate wash primer, which failed catastrophically after just a few cycles. Figure 32 shows these panels after being removed from the test chamber.

Table 17 ASTM D1654 ratings for GM9540P testing on multimetal specimens

		Exposure Time (cycles)							
		Creep from Scribe				Blisters in Field			
Pretreatment		20	40	60	80	20	40	60	80
Picklex 20	53022	3.2	0.8	0.4	0	4.2	3.2	1	0
	53030	8	7.8	7.4	6.8	8.6	8.2	7.6	7
SurTec 650	53022	4.8	3.6	2.6	1.6	6.6	6.2	4.2	3
	53030	7.2	6	6	6	9.2	8	6.8	6.4
Aero-Green AC-10	53022	6.2	5.4	5	4.8	8.2	6.8	5.8	5.8
	53030	8	6.4	6.2	6	10	10	8.8	8.8
PPG 11-TGL-07-Z	53022	6.4	5.4	4.8	4.8	9.4	7.8	6.4	6.2
	53030	8	6.4	6.4	6.4	10	8.6	8	7.2
Oxsilan 9810/2	53022	7	6.4	5	5	8.4	7.4	6.6	6.6
	53030	8.8	8	8	7.6	10	10	8.2	8.2
Bonderite 7400	53022	7.6	6.8	5.2	5	8.4	7.6	6.6	6.4
	53030	8.2	7	7	6.8	10	9.6	9.2	8.8
Ecosil Eco 5-1	53022	5.2	4.2	3.2	2.2	7	7	6	3.6
	53030	8.4	7.2	7.2	7.2	9.8	9.4	8.6	8.6
NCP WP N-8237-2.5 A/B	53022	0	0	0	0	0	0	0	0
	53030	7.4	5.2	4.8	4	9.2	8.4	7	5.8
DOD-P-15328 WP	53022	5.6	4.2	3	2	9.8	8.8	8	5.4
	53030	8.6	7.4	7.2	7.2	10	10	9.8	9.8
Direct to Metal	53022	6.2	4.8	3.6	3	9.2	8	5.6	5
	53030	8.4	6.6	6.6	6.4	9.2	6.2	5.6	5.6

Fig. 32 NCP nonchromate wash primer multimetal panels delaminating after 3 cycles

Figures 33–38 show the multimetal galvanic test specimen after 80 cycles. One can clearly see the superior corrosion protection provided by the MIL-DTL-53030 type IV versus the MIL-DTL-53022 type II primer. Examining only the MIL-DTL-53022 type II specimens, it is evident that there is less blistering along the scribes of the Bonderite M-NT-7400 specimens than on the baseline wash primer or the Ecosil 5-1 specimens. However, there is noticeably more delamination at the aluminum "L" bracket for Bonderite M-NT-7400 and Ecosil 5-1 than is seen on the baseline wash primer. Note that the MIL-DTL-53030 shows no delamination at the aluminum angle bracket.

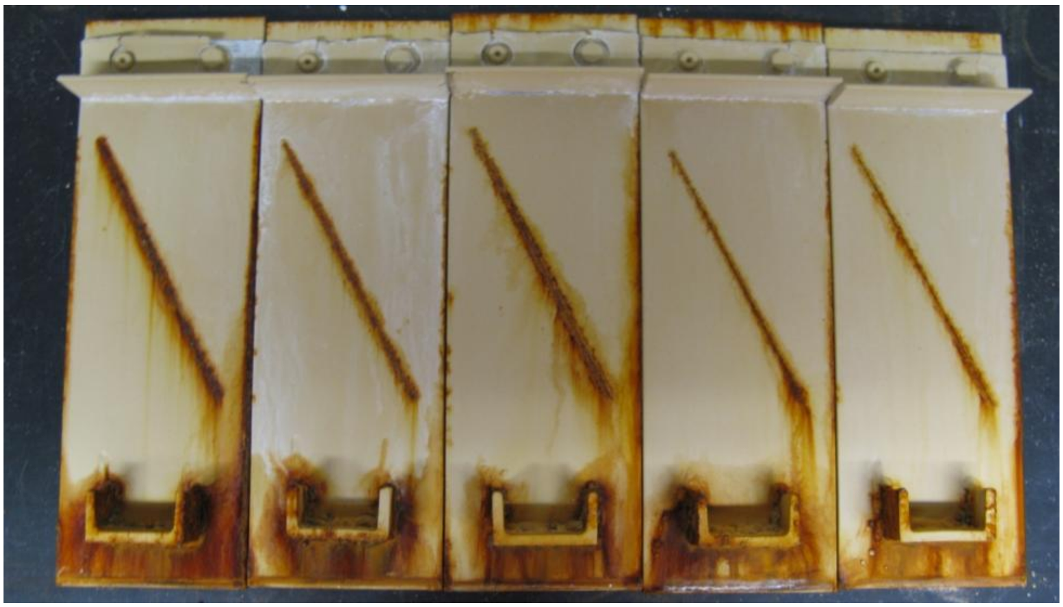


Fig. 33 Bonderite M-NT-7400 on multimetal panels primed with MIL-DTL-53022 after 80 cycles

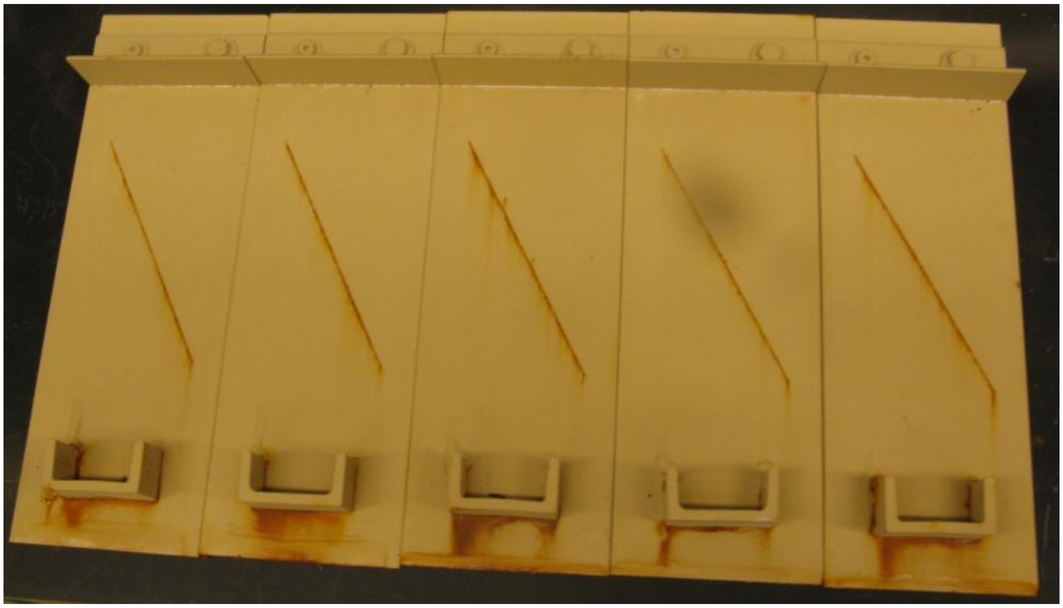


Fig. 34 Bonderite M-NT-7400 on multimetal panels primed with MIL-DTL-53030 after 80 cycles

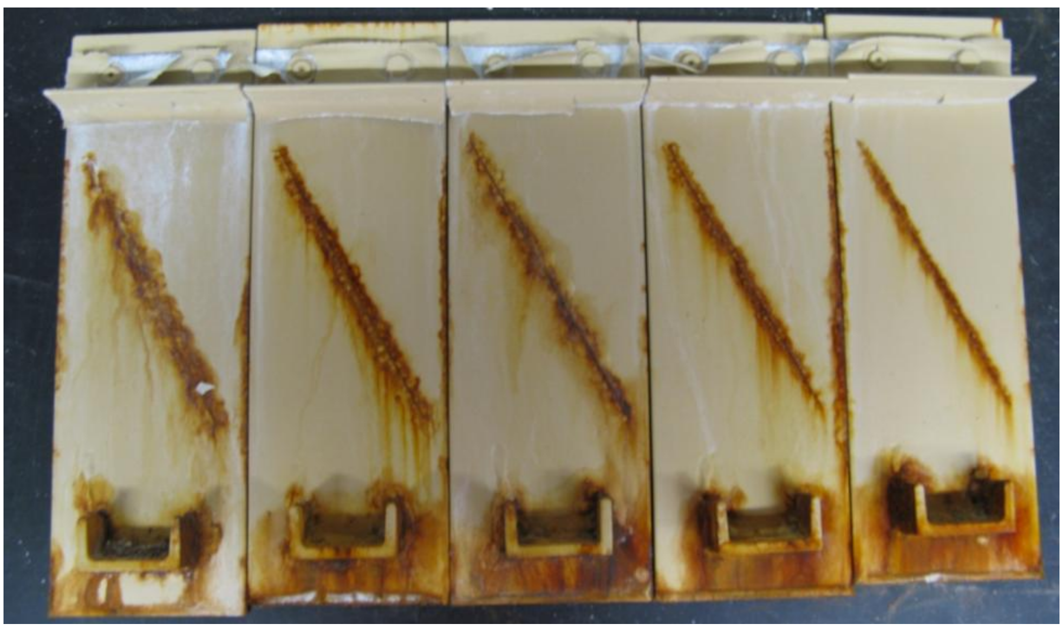


Fig. 35 Ecosil 5-1 on multimetals panel primed with MIL-DTL-53022 after 80 cycles

Fig. 36 Ecosil 5-1 on multimetals panel primed with MIL-DTL-53030 after 80 cycles

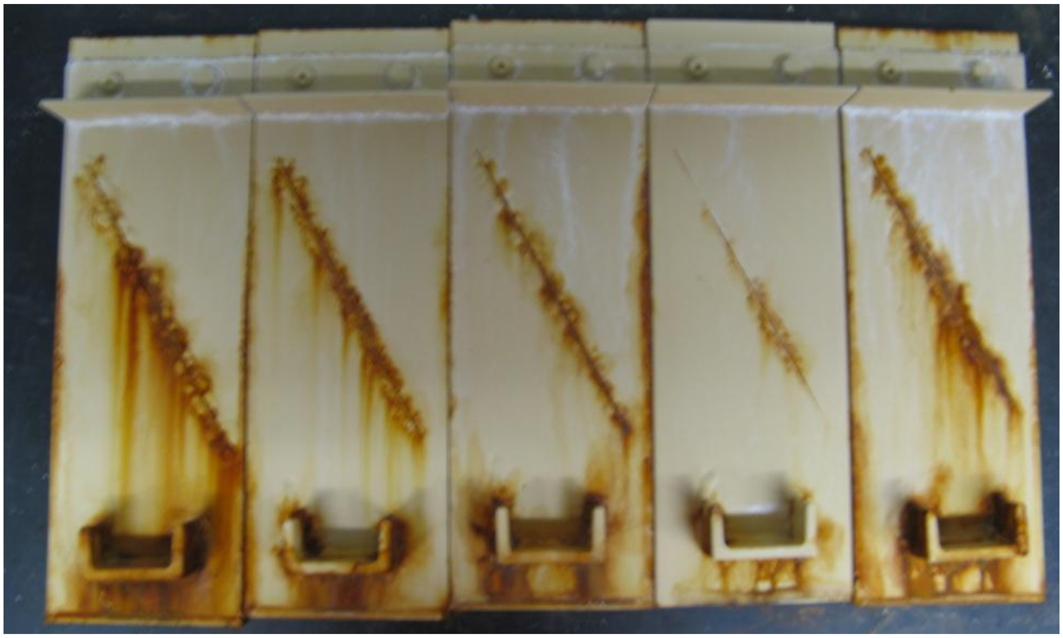


Fig. 37 DOD-P-15328 on multimetal panels primed with MIL-DTL-53022 after 80 cycles

Fig. 38 DOD-P-15328 on multimetal panels primed with MIL-DTL-53030 after 80 cycles

3.1.7 Outdoor Exposure

Outdoor exposure test coupons of aluminum, CRS, and multimetal assemblies were initiated at CCAFS on September 26, 2013. The success criteria are evaluated after 2 years of exposure at CCAFS, as is the case in qualification to TT-C-490. Using ASTM D1654 ratings, success is determined similarly to the accelerated corrosion chamber tests. CRS substrates must achieve rating ≥6 for creep from scribe and ≥7 for blisters in field. The aluminum substrates rating shall be ≥8 for creep from scribe and ≥7 blisters in field. The multimetal specimens rating shall be ≥6 for the scribed area and for area away from the scribe, ≥7 combined rating for blisters in field.

Table 18 contains all ratings for CRS taken at 6-month intervals. After 2 years of outdoor exposure, only 2 pretreatments, Bonderite M-NT-7400 and the NCP nonchromate wash primer, were able to meet the success criteria with both MIL-DTL-53022 and MIL-DTL-53030. The Bonderite M-NT-7400 and NCP also rated a 10 for blisters in field, far exceeding the required rating of 7. The consistent performance of the Bonderite M-NT-7400 in laboratory tests indicated that it would perform well in outdoor exposure. The NCP nonchromate wash primer provided good corrosion performance in outdoor exposure but was unable to meet minimum requirements in laboratory tests.

Table 18 ASTM D1654 ratings for outdoor exposure testing on CRS panels

Pretreatment		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	10	3.4	3	1.8	10	6.8	5.4	4
	53030	7.8	4.6	3.2	3.2	8	8	8	8
SurTec 650	53022	10	3	2.2	1.2	10	10	10	10
	53030	10	6	5	4.6	10	10	10	10
Aero-Green AC-10	53022	9.6	5.2	3.2	2.8	10	9.4	9.4	9
	53030	10	4.8	3.6	3.4	10	9.4	9.4	9.4
PPG 11-TGL-07-Z	53022	10	4	3.7	2.7	10	10	10	10
	53030	10	6.5	5.75	4.5	10	10	10	10
Oxsilan 9810/2	53022	10	0.8	0.8	0.5	10	10	10	10
	53030	10	6.6	3	3	10	10	10	10
Bonderite 7400	53022	10	8.6	7.3	7.3	10	10	10	10
	53030	10	8.6	7.0	6.4	10	10	10	10
Ecosil Eco 5-1	53022	10	4.5	3.5	3.3	10	9.6	9.6	9.4
	53030	10	8	7.6	6.8	10	9.6	9.6	9.6
NCP WP N-8237-2.5 A/B	53022	10	7.6	6.8	6.8	10	10	10	10
	53030	10	7	6	6	10	10	10	10
DOD-P-15328 WP	53022	10	4.4	3.4	2.2	10	10	10	10
	53030	10	5.8	3	2.6	10	10	10	10

More surprising was the inadequate performance of the baseline wash primer on CRS. The baseline wash primer did not meet the success criterion for creep from scribe after only 1 year of exposure. This was not in agreement with much of the baseline wash primer's performance on CRS in accelerated corrosion tests.

Figures 39–42 are presented as a visual comparison of the Bonderite M-NT-7400 versus the baseline wash primer on CRS test panels. These panels do not have an abrasive blasted profile and are a relatively smooth milled finish. Bonderite M-NT-7400 had markedly better performance than the baseline with MIL-DTL-53022. Although performance was also better with MIL-DTL-53030, there is far more undercutting of the coating along the scribe on the baseline wash primer samples.

Fig. 39 Bonderite M-NT-7400 on CRS primed with MIL-DTL-53022 after 2 years

Fig. 40 Bonderite M-NT-7400 on CRS primed with MIL-DTL-53030 after 2 years

Fig. 41 DOD-P-15328 on CRS primed with MIL-DTL-53022 after 2 years

Fig. 42 DOD-P-15328 on CRS primed with MIL-DTL-53030 after 2 years

In general, many of the pretreatments preformed adequately throughout the 2 years of outdoor exposure on aluminum substrates. The exception was the Picklex 20. The coating system on Picklex 20 samples with MIL-DTL-53022 completely delaminated from the substrate after 1 year (Fig. 43). All of the ratings for aluminum panels are presented in Table 19. Although all of the pretreatment products did not meet the success criteria of ≥8 for scribed area and ≥7 for blisters

in field, many did provide at least some benefit. The Bonderite M-NT-7400, a consistent performer thus far, barely missed meeting the success criteria with a 7.8 rating for scribed areas with MIL-DTL-53022, but exceeded the requirement with MIL-DTL-53030 and for blisters in field. The Bonderite product rated a 7.8 after 1 year but did not degrade any further through year 2. It can be argued that the Bonderite M-NT-7400, with standard error factored in, could be considered met in this case.

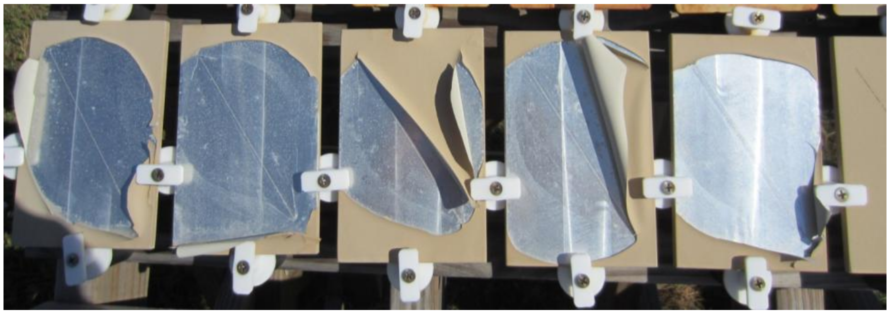


Fig. 43 Picklex on aluminum primed with MIL-DTL-53022 after 1 year

Table 19 ASTM D1654 ratings for outdoor exposure testing on aluminum panels

		Exposure Time (hours)							
		Creep from Scribe				Blisters in Field			
Pretreatment		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	8	0	0	0	10	0	0	0
	53030	10	7.2	6.6	5.6	10	6.8	6.2	4
SurTec 650	53022	10	9	9	9	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Aero-Green AC-10	53022	10	8.2	7.8	7.2	10	9.4	8.6	7.2
	53030	10	8.3	6.8	5.3	10	9.4	8	5.8
PPG 11-TGL-07-Z	53022	10	9	9	9	10	10	10	10
	53030	10	8.8	8.8	8.8	10	10	10	10
Oxsilan 9810/2	53022	10	9	9	9	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Bonderite 7400	53022	10	7.8	7.8	7.8	10	10	10	10
	53030	10	9	9	9	10	10	10	10
Ecosil Eco 5-1	53022	10	9	8.6	8.4	10	9.6	9.6	8.2
	53030	10	9	9	9	10	9.6	9.4	8.6
NCP WP N-8237-2.5 A/B	53022	10	8.6	8.6	8.4	10	10	10	10
	53030	10	9	9	9	10	10	10	10
DOD-P-15328 WP	53022	10	8.8	8.8	8.8	10	10	10	10
	53030	10	9	9	9	10	10	10	10

Figures 44–47 are presented as a visual comparison of the Bonderite M-NT-7400 versus the baseline wash primer on aluminum test panels. Small areas along the scribe were detected with the recital and measured to arrive at the ratings. However, no significant difference can be discerned visually.

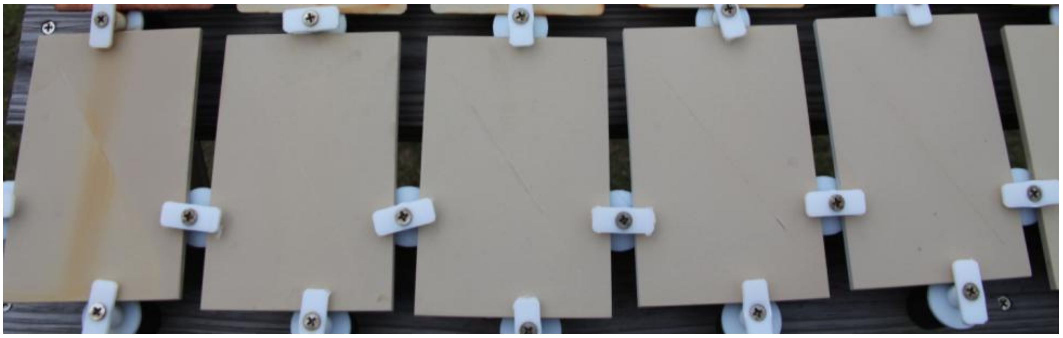


Fig. 44 Bonderite M-NT-7400 on aluminum primed with MIL-DTL-53022 after 2 years

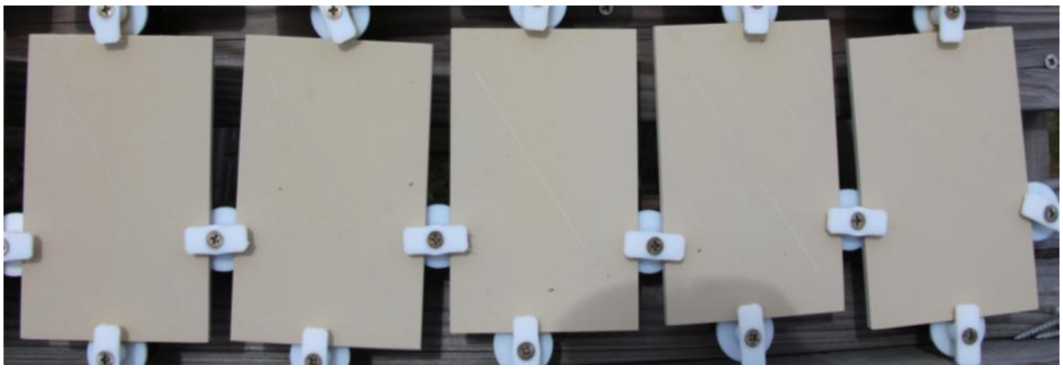


Fig. 45 Bonderite M-NT-7400 on aluminum primed with MIL-DTL-53030 after 2 years

Fig. 46 DOD-P-15328 on aluminum primed with MIL-DTL-53022 after 2 years

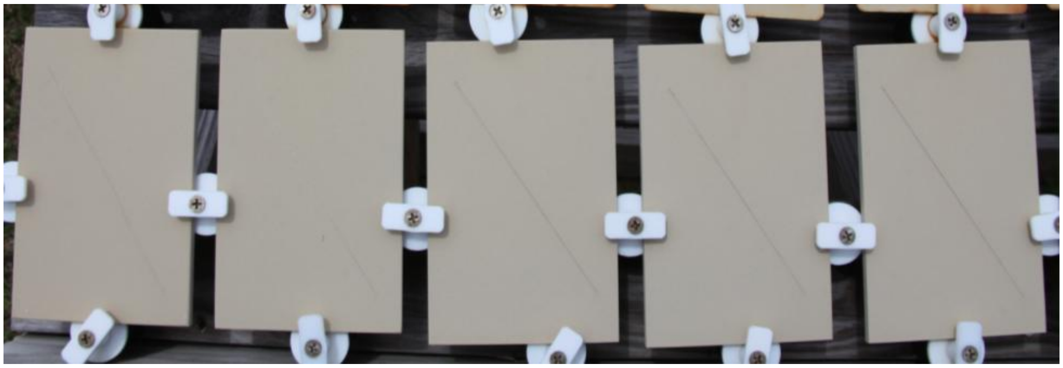


Fig. 47 DOD-P-15328 on aluminum primed with MIL-DTL-53030 after 2 years

Overall, the multimetal panels performed well in outdoor exposure regardless of the galvanic couples created by the welds, fasteners, and the extruded aluminum bracket. In general, if coating delamination occurred, it tended to be on the aluminum “L” brackets. Several of the NCP nonchromate wash primer panels also had delamination along the weld lines. This is reflected in the blisters in field ratings in Table 20. Some blistering was common in and on the U-weld channel, which was designed in such a manner so as to collect water. At 2 years, the baseline wash primer was the only pretreatment unable to meet the success criterion for creep from the scribe when primed with MIL-DTL-53030. With MIL-DTL-53022, the baseline wash primer as well as the Oxsilan 9810/2 could not meet the success criterion for the scribed area. However, the baseline was able to meet the criterion for blisters in the field. After examining all the results, we determined that the Bonderite M-NT-7400 is a viable replacement for chromate wash primer DOD-P-15328. The Bonderite M-NT-7400 has shown comparable performance in virtually all testing, has exceeded the performance of the baseline wash primer, and has met the success criteria in most cases.

Table 20 ASTM D1654 ratings for outdoor exposure testing on multimetal panels

Pretreatment		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
		0.5	1	1.5	2	0.5	1	1.5	2
Picklex 20	53022	8	7.8	7.8	7.4	10	8	7.2	7.2
	53030	9.8	8.8	8	7.3	10	8	6.5	6.5
SurTec 650	53022	10	9	7.8	7	10	8	6	6
	53030	9.8	9	7.2	6.8	10	8	6.2	6.2
Aero-Green AC-10	53022	7	6.8	6.8	6.8	10	8	6.6	6.6
	53030	10	8.6	8	7.2	10	8	7	7
PPG 11-TGL-07-Z	53022	9.6	8.8	7.8	6.6	10	8	7	7
	53030	10	9	7.6	6.4	10	8	7	7
Oxsilan 9810/2	53022	9.2	8.2	6	5.4	10	8	6.4	6.4
	53030	9.4	9	7.6	6.8	10	8	7	7
Bonderite 7400	53022	10	8.8	8.4	7.4	10	8	7.4	7
	53030	10	9	9	7	10	8	7.8	7
Ecosil Eco 5-1	53022	8.8	8.6	8.6	6.4	10	8	7	7
	53030	10	9	8.6	6.6	10	8	7.4	7
NCP WP N-8237-2.5 A/B	53022	8.4	8	7.8	6.6	10	8	7.2	6.2
	53030	8.6	8.4	8.4	7.8	10	8	6.6	6.6
DOD-P-15328 WP	53022	9.4	8.8	6.2	5.8	10	8	7.8	7
	53030	10	8.2	4.8	4.8	10	8	8	7
Direct to Metal	53022	10	9	7.6	6.4	10	8	6.8	6.8
	53030	9.6	8.8	7.8	6.6	10	8	7.8	7

3.1.8 Hydrogen Embrittlement

All of the Type 1d specimens were inspected under a 25× optical microscope after the requisite 150 h. No cracking was detected in any of the specimens tested. The ATC hydrogen embrittlement testing report is in Appendix C.

3.2 Demonstrations

3.2.1 Mine-Resistant Ambush-Protected (MRAP) Door Preparation

Two spare MRAP doors were obtained from the program manager's office and processed at their paint facilities at Aberdeen Proving Ground (APG), Maryland. Once processed, the doors were placed in local outdoor exposure testing at APG. Each door had the window masked off and was abrasive blasted to a 1.5-mil finish before processing. One door was treated with the baseline wash primer DOD-P-15328 and the other was treated with Oxsilan 9810/2. The Oxsilan 9810/2 was selected for demonstration based on early experimental results and the results leveraged from Environmental Security Technology Certification Program (ESTCP) project WP200906.[22] A representative from Chemetall, the manufacturer, was present for the Oxsilan pretreatment process, and the following procedures were followed:

Chemetall Oxsilan 9810/2:

1) Pressure washed all parts to remove dirt and grime.

2) Abrasive blasted to 1.5 Surface Profile IAW SSPC SP 10.

3) Blew-down dust.

4) Applied Oxsilan 9810/2 at 70–80 °F.

5) Allowed 60–90 s contact time.

6) Rinsed with clean deionized (DI) water and blown dry.

7) Applied CARC system after complete dry.

After pretreatment is fully dry, the doors were primed and painted side-by-side with MIL-DTL-53022 type II primer and top-coated with MIL-DTL-53039. Once the CARC system was cured, the doors were transported to ARL's Rodman Laboratory, Building 4600, where they were placed outside on exposure test racks beginning in August of 2012 (Fig. 48). This outdoor exposure site lacks an individual weather station, so specific weather data were not collected. However, the corrosion rate is approximately 0.04 mpy. APG can provide weather data when requested.

Fig. 48 MRAP door being treated with Oxsilan 9810/2 (left); painted doors on outdoor exposure racks at ARL (right)

The Oxsilan 9810/2 performed well in laboratory tests as part of the ESTCP program WP 200906 and was a major reason for considering it as a potential alternative to DOD-P-15328. Although not considered a full-scale demonstration, the MRAP doors provide some insight into the processing of larger parts in a production environment. Unlike the wash primer, this product requires a rinsing step following a dwell or contact period to remove the surplus product. In a small-scale repair scenario, this may not be practical. It is more appropriate for larger-scale production where a recirculating system can be employed and waste product is captured and reused. Nevertheless, Oxsilan 9810/2 has demonstrated good corrosion resistance and adhesion for abrasive-blasted steel substrates. Figures 49

and 50 show the 2 MRAP doors after 2 years of outdoor exposure at APG. Understandably, APG is not as aggressive as Cape Canaveral; however, the relative performance of the Oxsilan 9810/2 versus the baseline wash primer can still be quantitatively measured. Table 21 lists the ASTM D1654 ratings over the past 2 years. There is no difference in the corrosion present on the doors or along the scribes so far. Exposure at APG will likely take longer for significant corrosion to occur.

Fig. 49 MRAP door with Oxsilan 9810/2 after 2 years with close-up of scribed area

Fig. 50 MRAP door with DOD-P-15328 WP after 2 years with close-up of scribed area

Table 21 ASTM D1654 ratings for outdoor exposure testing on MRAP doors

		Exposure Time (years)							
		Creep from Scribe				Blisters in Field			
Pretreatment		0.5	1	1.5	2	0.5	1	1.5	2
Oxsilan 9810/2	53022	10	9	9	9	10	10	10	10
DOD-P-15328 WP	53022	10	9	9	9	10	10	10	10

3.2.2 Demonstrations on M200A1 Generator Trailers and Tricon Containers

LEAD provided 4 M200A1 generator trailers and 3 containers (tricons) from excess equipment set for disposal. LEAD was selected as the demonstration site due to LEAD's function as a repair and support facility. In addition, 2 aluminum 6061 panels were bolted to each of the 3 containers to create a galvanic couple. The assets were abrasive blasted to a 1.5-mil surface profile by LEAD personnel using a 30 × 60 garnet media. Bonderite M-NT 7400, Oxsilan 9810/2 Ready-to-Use (RTU), and PPG 11-TGL-02 (a modified formulation of the PPG Zircobond 4200) were selected as the candidates for application. One M200A1 trailer and one tricon container were pretreated with one of the candidate products by LEAD personnel with guidance from the manufacturer. The demonstration assets are as follows:

- Henkel Bonderite: one TRICON and one M200A1 Trailer
- Chemetall Oxsilan: one TRICON and one M200A1 Trailer
- PPG 11-TGL-07-Z: one TRICON and one M200A1Trailer

An additional M200A1 trailer was masked down the center to treat one-half with Bonderite M-NT 7400 and one-half with the baseline DOD-P-15328. After pretreatment applications, the trailers and containers were coated with MIL-DTL-53022 Type III CARC primer and MIL-DTL-53039 Type IX CARC topcoat by LEAD personnel. Once complete, the trailers and containers were placed in local outdoor exposure testing at LEAD. The half Bonderite, half wash primer trailer was sent to CCAFS for outdoor exposure, where it was placed 170 m from the ocean.

Henkel Bonderite M-NT 7400

The M-NT 7400 was shipped to LEAD in a concentrate form and was mixed on site by Henkel technicians. The Henkel product concentrate was diluted to a 5% solution using DI water provided by Henkel. The 7400 needs only to wet the substrate to have the desired reaction and was applied with a traditional high-volume, low-pressure (HVLP) spray gun with a 1.8-mm needle and 2-gal pressure pot supply. Any puddling of the pretreatment was blown off with compressed air. No post-rinse is required, which allows the M-NT 7400 to be applied in standard paint booths at LEAD. The temperature over the course of application was 70 °F, and the humidity ranged from 58% to 81%. The paint booths at LEAD have heaters for paint curing and were used for 30 min at 120 °F to accelerate the drying of the pretreatment.

The LEAD personnel tasked with application noted that the Bonderite M-NT 7400 was an easy-to-use product and appreciated that they could continue to use their current HVLP spray gear. One LEAD applicator commented that the process “fit like a glove” because it was similar to wash primer application. All assets were coated with the M-NT 7400 in less time than it took to coat an equivalent number of assets with wash primer. Only 2 L of the M-NT 7400 were required for full coverage of the assets. The Bonderite M-NT 7400 has no “pot life” limits compared to the DOD-P-15328 wash primer, which has a 4-h pot life. The pot life refers to the amount of time the applicators have to use the product before it has polymerized beyond use. With a typical wash primer, once 4 h has passed, any unused wash primer must be disposed of as hazardous waste. By contrast, the Bonderite M-MT 7400 can be left in the spray gun (pot) for the next shift to use.

Figure 51 shows photos of the container before and after the Bonderite M-NT 7400 treatment. The container and trailer treated fully coated can be seen in Fig. 52. In

addition to these 2 assets, the split trailer mentioned previously was treated at this time.

Fig. 51 Abrasive blasted tricon before (left) and after (right) Bonderite M-NT 7400

Fig. 52 M200A1 trailer with split pretreatments (left) and with full CARC system (right)

Chemetall Oxsilan 9810/2 RTU

The Oxsilan 9810/2 was delivered to LEAD as a ready-to-use solution. The Oxsilan product requires a post-rinse with DI water following a 60- to 90-s dwell time. The required post-rinse necessitated the use of a wash bay at LEAD to apply and rinse the Oxsilan 9810/2 (Fig. 53). LEAD safety personnel cleared the Oxsilan to enter the waste stream from the wash bay, and DI water had to be brought on-site for the demonstration as it is not available in the facility. The DI came in a 55-gal drum, and Chemetall representatives added 200 mL of their flash-rust prevention additive 9943 to the rinse water. A pump was provided by Chemetall to maintain the required flow rate of material between 4 and 5 gallons per minute (GPM) (Fig. 54). After application and dwell, the assets were rinsed in the DI water/additive mix and blown dry with shop air. The air drying process took approximately 10 min for both the tricon and trailer. Ambient temperature ranged from 62 °F to 72 °F and humidity

ranged from 81% to 100% over the course of application. Once fully dry, the assets were moved to the in-line paint booth for the application of the CARC system. A photo of the assets in outdoor exposure at LEAD can be seen in Fig. 55.

Fig. 53 Wash bay at LEAD (left) and M200A1 trailer (right) treated with Oxsilan 9810/2 showing what appears to be slight flash rust in some areas

Fig. 54 GPM pump provided by Chemetall for application of Oxsilan 9810/2

Fig. 55 Painted assets deployed in outdoor exposure testing at LEAD

Following the application of the Oxsilan 9810/2, it was observed that, regardless of the 9943 additive, areas of the M200A1 trailer showed some signs of flash rusting.

It was likely because only one applicator was used, and the more complex geometry of the trailer made it more difficult to monitor all of the surface areas during processing. The Oxsilan 9810/2 also required a dwell time, a post-rinse, and a special pump to maintain 4–5 GPM. This was a deviation from the conventional wash primer process, which is dry in place and uses a standard HVLP spray gun. The dwell and rinse would require additional time for the processing of equipment versus the conventional wash primer. However, like the Bonderite product, there is no listed pot life for Oxsilan 9810/2, and it would significantly reduce the amount of hazardous waste created by the disposal of unused wash primer.

PPG 11-TGL-27

This product was provided by PPG in place of the previously tested Zircobond 4200. PPG indicated that the 11-TGL-27 was a reformulation of the 4200 specifically geared toward spray application. The 11-TGL-27 was provided ready-to-use by PPG and was agitated prior to application using a squirrel cage mixer attached to a portable drill. Application was performed using a standard HVLP spray gun. A 5-min dwell time was required before an initial post-rinse using the facility tap water, followed by a final post-rinse in DI water (provided by PPG). The assets were blown dry with shop air, with particular attention given to the areas susceptible to puddling. As the assets dried, evidence of flash rusting was observed, which can be seen in Fig. 56. Figure 57 shows the results of a tape pull test on the treated M200A1 trailer. LEAD personnel continued to paint the assets; however, it was decided that if the pretreatment appears to be causing flash rust, the demonstration would be considered a failure and no further observations would be made. Regardless of the performance results of the PPG 11-TGL-27, it would be difficult to persuade a user to adopt a process that leaves even an appearance of flash rusting to the degree that was observed here.

Fig. 56 M200A1 trailer during treatment (left) and container dry following treatment (right) with PPG 11-TGL-27

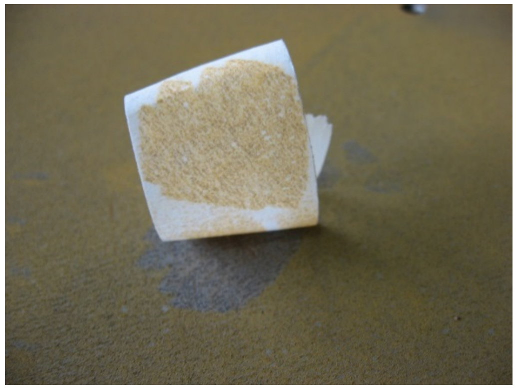


Fig. 57 Result of masking tape pull on dried PPG 11-TGL-27 treated assets

After allowing 2 weeks for a full cure, ARL returned to LEAD to perform dry film measurements and adhesion tests. Pull-off adhesion was performed on the M200A1 trailers in accordance with ASTM D4541, while crosshatch adhesion in accordance with ASTM D3359 was used on all assets. Dry film thickness was measured using a Tooke gauge. All of these methods are destructive evaluation methods, therefore all areas were repaired after testing by LEAD personnel using MIL-DTL-53022 Type III. An example of this can be seen in Fig. 58.

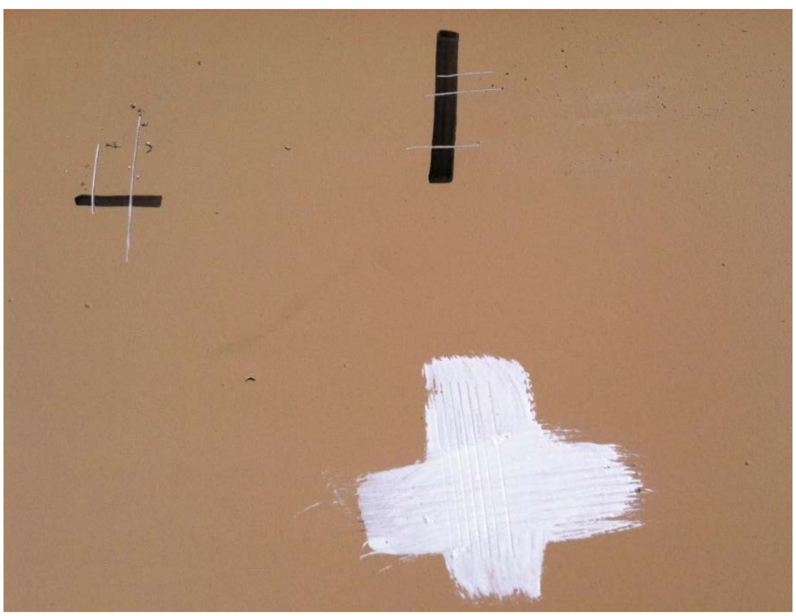


Fig. 58 Tooke and crosshatch tests on asset

For ASTM D4541, a total of 4 pulls were made on each trailer. The pull-off values were averaged and are presented in Fig. 59. Only the baseline wash primer exceeded 800 psi for pull-off strength. Pull-off strength is not a requirement as per TT-C-490 and is only included for comparison. All systems performed adequately and no adhesion issues were observed as a result of pretreatment issues. The mode

of failure was cohesive failure of the topcoat for all pulls made, and not at the primer/substrate interface. This is an indication that the top coat was not fully cured and not indicative of pretreatment failures. The pretreatments did not contribute to the pull-off failures in any system.

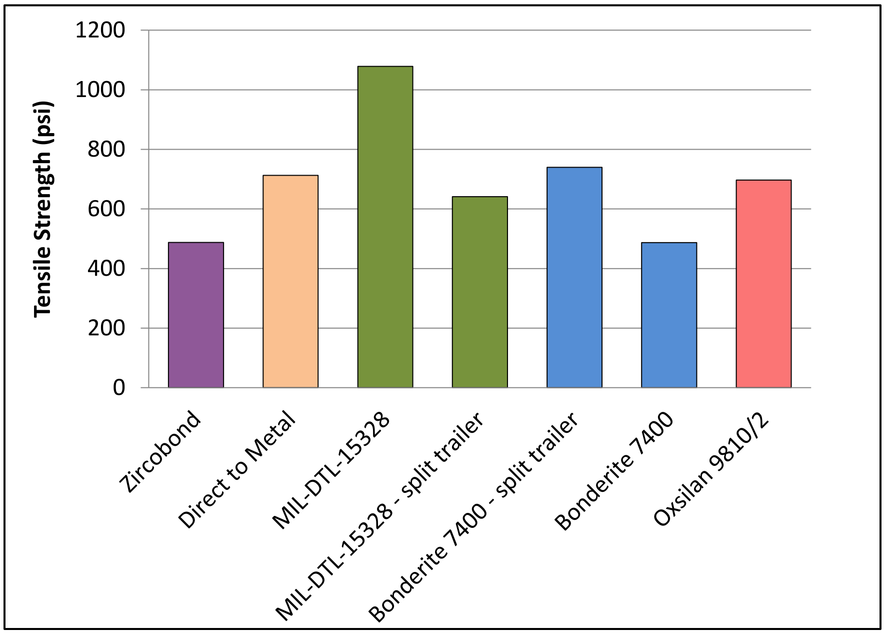


Fig. 59 Pull-off adhesion results from LEAD M200A1 trailers

A minimum rating requirement for crosshatch dry tape adhesion is 4.0 per TT-C-490. The results shown in Table 22 represent the average of 4 tape pulls. All of the pretreatment systems tested were able to achieve a 4 or better, meeting the minimum requirement.

Table 22 Ratings from dry tape adhesion tests

	Cross Hatch Ratings
Bonderite Tricon	4.5
Bonderite Trailer	4.5
Bonderite Half Trailer	4.5
Bonderite Panel	5
Oxsilan Tricon	4.75
Oxsilan Trailer	4.25
Oxsilan Panel	4
Wash Primer Tricon	4
Wash Primer Trailer	4.5
Wash Primer Half Trailer	5
Wash Primer Panel	4

Two measurements were taken on each asset and averaged using the Tooke dry film gage. The average film thicknesses are shown in Table 23. While there is some variation, this would be expected at depot-level maintenance. Typical depot or even original equipment manufacturer (OEM) painting on complex geometries such as these assets presents many areas whereby a spray painter has to negotiate changes in painting direction on appurtenances and corners of fenders, hitches, and other obstacles. Unlike painting flat test coupons, an asset painter must also maintain a "wet-edge" so that the final paint film is continuous and free of dry spray. This activity will commonly result in variations in dry film thickness (DFT), which are acceptable IAW MIL-DTL-53072. There is a 38% difference between the thickest and the thinnest coatings across all of the assets measured in the demonstration. For the reasons stated previously, this is not considered unusual or significant on OEM manufactured assets or at this level of maintenance. To ensure that edges and shadowed surfaces are adequately coated, painters may apply additional paint to adjacent areas. Therefore, additional DFT in these areas is common and not unexpected. The thickness measurements in Table 23 were deemed satisfactory.

Table 23 Dry film thickness measurements of LEAD demonstration assets

Asset	Pretreatment	Average Tooke Dry Film Thickness Measurement		
		Primer	Topcoat	Total
M299A1 Trailer	No Pretreatment	2.0	2.8	4.8
Tricon Container	DOD-P15328	1.7	2.8	4.5
M299A1 Trailer	DOD-P-15328	3.0	2.5	5.5
Tricon Container	Bonderite N-MT 7400	2.5	3.0	5.5
M299A1 Trailer	Bonderite N-MT 7400	1.8	4.5	6.3
Tricon Container	Oxsilan 9810/2	2.5	2.0	4.5
M299A1 Trailer	Oxsilan 9810/2	3.8	3.5	7.3
Split Pretreatment M200A1 Trailer	DOD-P-15328	3.0	3.0	6.0
	Bonderite N-MT 7400	2.0	3.0	5.0

The M200A1 trailer prepared at LEAD to be half wash primer, half Bonderite M-NT 7400 was deployed to the CCAFS Outdoor Exposure Site on June 7, 2015. The trailer had its tires deflated and was set on concrete blocks to support it, and then tied down with chains to prevent any migration in the event of tropical storms or hurricanes. A photograph of this initial state can be seen in Fig. 60. During the 2-year exposure period, the trailer was subject to 2 separate tropical storms as well as Hurricane Matthew in 2016. The photo in Fig. 61 was taken on June 7, 2017, exactly 2 years after deployment. Readily apparent in the photographs is the distinct lack of corrosion on either side of the trailer. Aside from some staining around the

bolts and faying surfaces, as well as peripheral rusting on the leading edge of the trailer, most of the trailer is corrosion free. Because the leading edge is showing similar issues on both sides, it is possible that this could be attributed to painter technique and shadowing. There are no signs of blistering under the coating anywhere, and the only measurable corrosion creep can be seen on the sites where crosshatch testing was performed (these spots were only repaired with a brush-on primer). The corrosion rate observed on mass loss coupons was 2.41 mpy during this period, about 0.6 mpy higher than the recorded average since 2010.

Fig. 60 Initial deployment of M200A1 trailer at Cape Canaveral

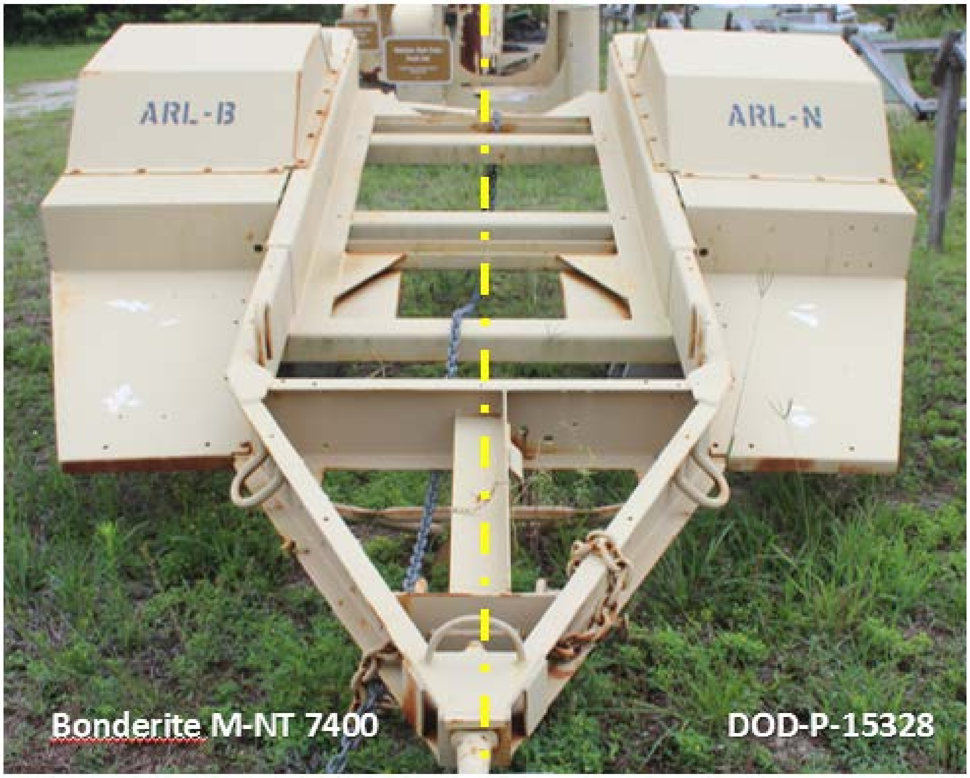


Fig. 61 Front of M200A1 trailer with split pretreatments after 24 months at Cape Canaveral: Bonderite (left) and wash primer (right).

3.2.3 Demonstration on M373A2 Trailers

Only the Bonderite M-NT 7400 was selected to be demonstrated against the baseline wash primer. A pair of M373A2 trailers was provided by LEAD for an additional demonstration. Unlike the previous demonstration, no guidance was provided by the manufacturers on site. The larger M373A2 trailers offered a variety of substrates ranging from wood to aluminum to steel with complex surface finishes and geometries. Figure 62 is one of the M373A2 trailers that has been prepared prior to pretreatment. Across all of these substrates remained areas that were still painted, often with several layers with correspondingly increased coating thicknesses. This is a typical scenario found in depot- and field-level maintenance.

Fig. 62 Prepped M373A2 trailer ready for pretreatment application

The wash primer was applied to one of the M373A2 trailers IAW DOD-P-15328, and Bonderite M-NT 7400 was spray-applied to another M373A2, ensuring that the exposed metal substrates were properly covered. During the course of application, there was intentional overspray onto the painted areas to detect any incompatibilities between the pretreatment and the painted surfaces. After pretreatment application, the trailer was blown dry with shop air. The Bonderite M-NT 7400 also was force dried using the booth ovens at 120 °F for 30 min. The booth oven drying accelerates the drying of the Bonderite M-NT 7400 that penetrates between faying surfaces. The LEAD personnel then primed and top coated the trailers with MIL-DTL-53022 Type III and MIL-DTL-53039 Type IX, respectively. LEAD personnel applied the same CARC system to both M373A2 trailers; however, ARL was not on site to witness this process.

Figures 63 and 64 show the right (top) and left (bottom) side of the 2 M373A2 trailers used in the demonstration. Each of the numbers in the photographs represent the spots where the adhesion tests were taken and recorded in Table 24. The numbers correspond with the "reading" numbers in each of the tables, which also show the type of surface beneath the pretreatment and CARC system.

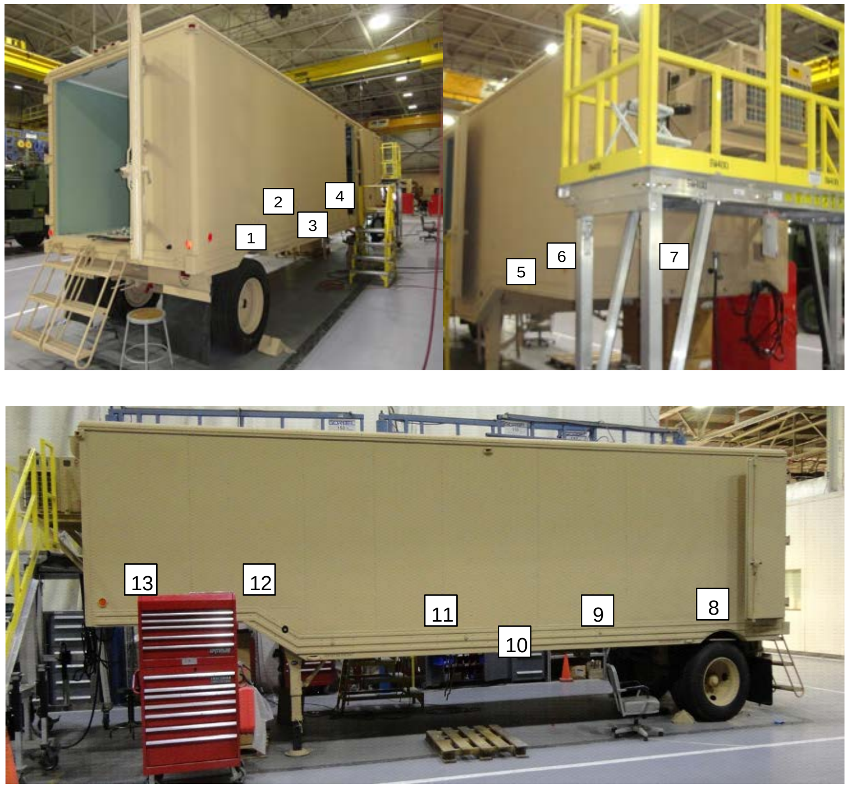
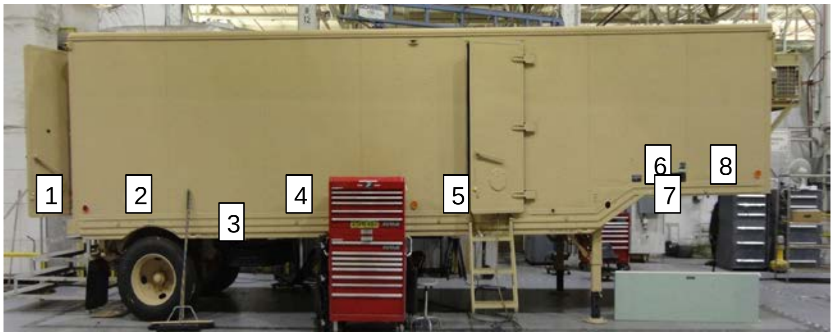


Fig. 63 The M373A2 trailer pretreated with baseline DOD-P-15328 wash primer and complete CARC system. The numbers indicate where adhesion tests were performed.



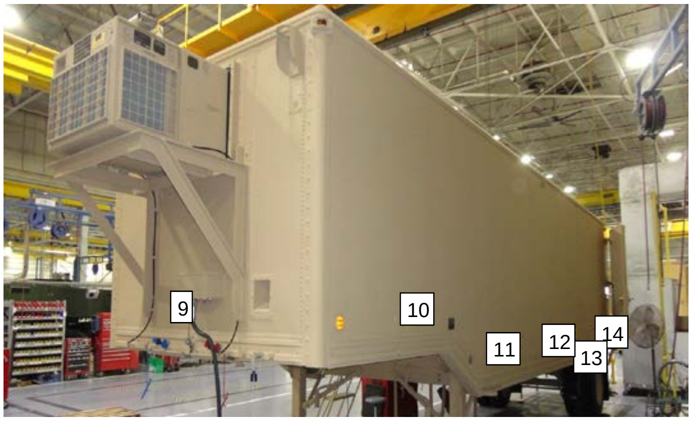


Fig. 64 The M373A2 trailer pretreated with Bonderite M-NT-7400 and complete CARC system. The numbers indicate where adhesion tests were performed.

Table 24 Crosshatch adhesion test results from areas of the M373A2 trailers

	Bonderite M-NT 7400			DOD-P-15328		
Reading	Location	DFT (mils)	Rating	Location	DFT (mils)	Rating
1	Panel	9.2	5	Panel	10.3	4
2	Panel	12.9	0	Panel	14	0
3	Base Frame	10.6	3	Base Frame	10.5	4
4	Panel	11.4	4	Panel	20.4	4
5	Panel	13.9	4	Panel	20.3	1
6	Panel	14.3	4	Panel	17.2	0
7	Base Frame	7.6	3	Panel	8.6	5
8	Panel	9.2	5	Panel	21.8	4
9	Panel	10.1	1	Panel	9.5	4
10	Panel	20.2	5	Base Frame	9.4	3
11	Panel	10.1	4	Panel	6.1	5
12	Panel	9.2	5	Panel	7.7	4
13	Panel	11.7	0	Panel	17.9	0
14	Base Frame	10.4	5			
Average		**11.5**	**3.4**		**13.4**	**2.9**
Std Dev		**3.1**	**1.8**		**5.5**	**1.9**

ARL returned to LEAD 2 weeks after the CARC application to perform crosshatch (dry) adhesion testing as per ASTM D3359 and to measure dry film thickness using the Tooke gage. Table 24 provides a side-by-side comparison of the crosshatch adhesion testing results from the M373A2 trailer pretreated with Bonderite M-NT 7400 versus the trailer pretreated with the baseline DOD-P-15328 wash primer. The average overall dry film thicknesses for both trailers are very similar but are thicker than allowed by MIL-DTL-53072. Adhesion tests of various areas of each trailer indicate that coating adhesion is very similar. The Bonderite M-NT 7400 provides slightly better adhesion than the DOD-P-15328 wash primer by about 17%. Overall, both pretreatments show very similar coating adhesion on identically prepared M373A2 trailers.

4. Conclusions

The results presented here show that there are viable alternatives that can provide comparable, and in some cases improved, performance to the legacy DOD-P-15328 chromated wash primer. The Bonderite M-NT-7400 is one of the products that compares very well with the baseline wash primer and, as demonstrated, proves to be a viable drop-in replacement. For all of the products tested, a deoxidizing step is advised to improve performance on aluminum substrates.

The demonstrations showed that the application process for the Bonderite M-NT-7400 is the most similar to wash primer application. It can be applied in a standard paint booth with common HVLP spray equipment. Application adjustments can easily be made on-the-fly. Pretreatment with Bonderite M-NT-7400 takes less time than with DOD-P-15328 wash primer and requires far less material. There is no prescribed pot-life for Bonderite M-NT-7400 resulting in little to no waste needing disposal. The favorable performance, application process, and environmental benefits support the Bonderite M-NT-7400 as a viable and more desirable option to the DOD-P-15328 chromated wash primer.

As tested, the DOD-P-15328 wash primer would not meet some of the requirements of TT-C-490F and would not be approved for use in many of the applications today. While it performed well in most accelerated corrosion tests, it did not meet the success criterion for creep from scribe after only 1 year of outdoor exposure. Perhaps this may facilitate discussion as to why nonchromate pretreatments are being required to meet a standard that is difficult for the legacy baseline wash primer to meet.

The Bonderite M-NT-7400 and other alternatives have been qualified for use as a replacement for chromated wash primer and are listed on the TT-C-490F QPD. Implementation of the alternatives is ongoing and will be expedited when the

DOD-P-15328 is cancelled. In October 2016 the CARC Commodity Manager of the Weapons and Materials Research Directorate (ARL) released a memo notifying the users of DOD-P-15328 of ARL's intent to cancel the wash primer specification on September 30, 2017. This will begin the transition toward full implementation of TT-C-490F type III and IV pretreatments on military systems that currently rely on DOD-P-15328.

5. References

1. US Department of the Army. Army materiel maintenance-maintenance of supplies and equipment. Washington (DC); 2013. Army Regulation No.: AR 750-1.
2. MIL-DTL-53072. Chemical agent resistant coating (CARC) system application procedures and quality control inspection. Washington (DC): US Department of Defense; 2003 June.
3. TT-C-490. Chemical conversion coatings and pretreatments for metallic substrates (base for organic coatings). Washington (DC): US Department of Defense; 2013 Jan.
4. MIL-DTL-53022. Primer, epoxy coating, corrosion inhibiting, lead and chromate free. Washington (DC): US Department of Defense; 2007 Jan.
5. MIL-DTL-53030. Primer coating, epoxy, water based, lead and chromate free. Washington (DC): US Department of Defense; 2010 May.
6. MIL-DTL-53039. Coating, aliphatic polyurethane, single component, chemical agent resistant. Washington (DC): US Department of Defense; 2005 June.
7. MIL-DTL-64159. Camouflage coating, water dispersible aliphatic polyurethane, chemical agent resistant. Washington (DC): US Department of Defense; 2002 Jan.
8. DOD-P-15328. Primer (wash), pretreatment (formula no. 117 for metals). Washington (DC): US Department of Defense; 1978 Apr.
9. Chesonis KG, Miller CE. VOC compliant wash primer. Aberdeen Proving Ground (MD): Army Research Laboratory (US); 1994 Sep. Report No.: ARL-MR-190.
10. Young JJ. Minimizing the use of hexavalent chromium (Cr6+). Washington (DC): Undersecretary of Defense; 2009.
11. ASTM A1008. Standard specification for steel, sheet, cold-rolled, carbon, structural, high-strength low-alloy, high-strength low-alloy with improved formability, solution hardened, and bake hardenable. West Conshohocken (PA): ASTM International; 2013.

12. ASTM A240/A240M-14. Standard specification for chromium and chromium-nickel stainless steel plate, sheet, and strip for pressure vessels and for general applications. West Conshohocken (PA): ASTM International; 2013.

13. FED-STD-141B. Federal test method standard: metals; test methods. Washington (DC): General Services Administration; 1967 Nov 24, 2014 Oct 17.

14. ASTM D3359. Standard test methods for measuring adhesion by tape test. West Conshohocken (PA): ASTM International; 2009.

15. ASTM D3363. Standard test method for film hardness by pencil test. West Conshohocken (PA): ASTM International; 2005.

16. SAE J400. Test for chip resistance of surface coatings. Reaffirmed: 2012-10-23.

17. ASTM B117. Standard practice for operating salt spray (fog) apparatus. West Conshohocken (PA): ASTM International; 2011.

18. GM 9540P. Accelerated corrosion test. Detroit (MI): General Motors Engineering Standards; 1997.

19. Corrosion tests and standards: application and interpretation. 2nd ed. West Conshohocken (PA): ASTM International; 2005. Chapter 30, Seawater; p. 363. Print.

20. Travel weather averages (Weatherbase). Weatherbase. N.p., n.d [accessed 2017 Sep 21]. http://www.weatherbase.com/weather/weather-summary.php3?s=49747&cityname=Cape+Canaveral%2C+Florida%2C+United+States+of+America&units=.

21. ASTM F519-13. Standard test method for mechanical hydrogen embrittlement evaluation of plating/coating processes and service environments. West Conshohocken (PA): ASTM International; 2013.

22. Kelley J, Braswell T. ESTCP Project 200906 non-chromate, ZVOC coatings for steel substrates on Army and Navy aircraft and ground vehicles: technology area 1: demonstration of steel pretreatments on high hard armor steel. Aberdeen Proving Ground (MD): Army Research Laboratory (US); 2014 Dec.

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Appendix A. Observed Corrosion Rate (mils/year) at Cape Canaveral Air Force Station (CCAFS) since 2011

Coupon	1	2	3	Avg	Date Exposed	Date Removed	Days Exposed	1	2	3	Avg	Δm	Corrosion Rate
660F	29.81	29.81	29.81	29.81	11/9/2010	5/17/2011	189	29.28	29.28	29.28	29.28	**0.53**	1.675323221
661F	29.89	29.89	29.89	29.89	11/9/2010	5/17/2011	189	29.40	29.40	29.40	29.40	**0.49**	1.560401741
662F	29.97	29.97	29.97	29.97	11/9/2010	5/17/2011	189	29.42	29.42	29.42	29.42	**0.55**	1.732256798
663F	29.94	29.94	29.94	29.94	11/9/2010	5/17/2011	189	29.48	29.47	29.48	29.47	**0.47**	1.48343598
664F	29.99	29.99	29.99	29.99	11/9/2010	12/20/2010	41	29.82	29.82	29.82	29.82	**0.17**	2.488413904
665F	29.91	29.91	29.91	29.91	11/9/2010	2/16/2011	99	29.59	29.59	29.59	29.59	**0.32**	1.952419132
666F	30.26	30.26	30.26	30.26	11/9/2010	5/17/2011	189	29.53	29.53	29.54	29.53	**0.73**	2.293157965
667F	30.07	30.07	30.07	30.07	11/9/2010	11/19/2012	741	27.27	27.27	27.27	27.27	**2.81**	2.26320573
668F	29.74	29.74	29.74	29.74	11/9/2010	11/19/2012	741	27.27	27.27	27.27	27.27	**2.47**	1.992675197
669F	29.80	29.80	29.80	29.80	11/9/2010	5/17/2011	189	29.06	29.06	29.06	29.06	**0.74**	2.343765589
670F	29.99	29.99	29.99	29.99	11/9/2010	2/16/2011	99	29.68	29.68	29.68	29.68	**0.31**	1.861842987
671F	30.02	30.02	30.02	30.02	11/9/2010	12/20/2010	41	29.85	29.85	29.85	29.85	**0.17**	2.493274088
296C	29.73	29.73	29.73	29.73	2/15/2011	2/23/2012	373	28.81	28.82	28.82	28.82	**0.92**	1.46699359
297C	29.75	29.75	29.75	29.75	2/15/2011	2/23/2012	373	28.66	28.66	28.66	28.66	**1.10**	1.755477399
442F	29.91	29.91	29.91	29.91	5/17/2011	11/19/2012	552	27.95	27.95	27.95	27.95	**1.96**	2.120827315
443F	29.70	29.70	29.70	29.70	5/17/2011	11/19/2012	552	28.19	28.19	28.19	28.19	**1.51**	1.639625135
513T	29.90	29.90	29.90	29.90	2/23/2012	2/11/2014	719	28.05.	28.05	28.05	28.05	**1.84**	1.531089586
514T	29.84	29.84	29.84	29.84	2/23/2012	2/11/2014	719	27.77	27.77	27.77	27.77	**2.07**	1.720795591
100S	29.93	29.93	29.93	29.93	11/19/2012	2/11/2014	449	28.74	28.74	28.74	28.74	**1.20**	1.59147734
101S	29.76	29.75	29.75	29.75	11/19/2012	2/11/2014	449	28.55	28.55	28.55	28.55	**1.20**	1.601684807
672F	30.05	30.05	30.05	30.05	11/9/2010	12/20/2010	41	29.91	29.91	29.91	29.91	**0.14**	2.104459415
673F	30.15	30.15	30.15	30.15	11/9/2010	2/16/2011	99	29.81	29.81	29.81	29.81	**0.34**	2.053059294
674F	29.97	29.97	29.97	29.97	11/9/2010	5/17/2011	189	29.33	29.33	29.33	29.33	**0.64**	2.028522264
675F	30.29	30.29	30.30	30.29	11/9/2010	11/19/2012	741	28.56	28.56	28.56	28.56	**1.73**	1.399444227
676F	30.03	30.03	30.03	30.03	11/9/2010	11/19/2012	741	28.23	28.23	28.23	28.23	**1.80**	1.450538463
677F	30.08	30.08	30.08	30.08	11/9/2010	5/17/2011	189	29.49	29.49	29.49	29.49	**0.59**	1.86299316
678F	30.01	30.01	30.01	30.01	11/9/2010	2/16/2011	99	29.66	29.65	29.65	29.65	**0.35**	2.143635439
679F	30.09	30.09	30.09	30.09	11/9/2010	12/20/2010	41	29.92	29.92	29.92	29.92	**0.17**	2.537015738
298C	29.61	29.61	29.61	29.61	2/15/2011	2/23/2012	373	28.75	28.75	28.75	28.75	**0.86**	1.373503467
299C	29.75	29.76	29.76	29.76	2/15/2011	2/23/2012	373	28.89	28.89	28.89	28.89	**0.86**	1.380982677
440F	29.88	29.89	29.89	29.89	5/17/2011	11/19/2012	552	28.50	28.50	28.50	28.50	**1.38**	1.498116316
441F	29.91	29.92	29.92	29.92	5/17/2011	11/19/2012	552	28.32	28.32	28.32	28.32	**1.60**	1.732400049
515T	29.89	29.89	29.89	29.89	2/23/2012	2/11/2014	719	27.72	27.72	27.72	27.72	2.17	1.8069878
516T	29.73	29.72	29.72	29.72	2/23/2012	2/11/2014	719	27.82	27.82	27.82	27.82	1.91	1.584717215
512T	29.92	29.92	29.92	29.92	2/23/2012	2/11/2014	719	27.74	27.74	27.74	27.74	2.17	1.806433509
519T	29.89	29.89	29.89	29.89	2/23/2012	2/11/2014	719	28.02	28.02	28.02	28.02	1.87	1.553676934
104S	29.82	29.82	29.82	29.82	11/19/2012	2/11/2014	449	28.47	28.47	28.47	28.47	1.36	1.804502751
105S	29.94	29.94	29.94	29.94	11/19/2012	2/11/2014	449	28.71	28.71	28.71	28.71	1.23	1.63674524
											Avg rate		**1.824365186**

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Appendix B. Weather Data for 2012–14 at Cape Canaveral Air Force Station (CCAFS)

2012–13

Row Labels	Sum of Rain, in	Sum of Solar Radiation, W/m²	Average of Temp, °F	Average of RH, %	Average of DewPt, °F	Average of Wetness, %	Sum of Time of Wetness, hr
Jan	0.00	375,267.70	62.6	76.4	54.2	56.1	503.3
Feb	0.92	435,153.20	68.6	79.9	61.6	58.0	479.5
Mar	0.83	742,881.40	73.5	74.2	64.1	60.6	509.8
Apr	0.74	801,708.10	75.6	69.5	63.9	51.6	446.8
May	2.96	819,811.80	79.7	77.4	71.4	58.5	547.0
Jun	6.80	692,588.30	80.9	79.7	73.5	57.0	556.8
Jul	3.58	878,208.60	83.5	82.6	77.3	65.8	654.3
Aug	6.67	675,046.90	81.9	86.8	74.9	74.8	734.5
Sep		634,962.40	81.1	81.9	70.6	74.7	720.0
Oct		508,110.60	76.7	77.5	69.6	68.6	744.0
Nov		353,967.40	65.9	80.7	67.1	69.3	715.8
Dec		298,045.20	66.5	83.0	60.6	80.5	706.8
Grand Total	22.50	7,215,751.60	74.7	79.1	67.4	64.7	7318.3

2013–14

Row Labels	Sum of Rain, in	Sum of Solar Radiation, W/m²	Average of Temp, °F	Average of RH, %	Average of DewPt, °F	Average of Wetness, %	Sum of Time of Wetness, hr
Jan	0.00	308,732.90	67.6	85.5	62.7	84.9	738.3
Feb	1.06	464,236.70	66.2	77.6	58.1	69.0	609.8
Mar	0.84	717,370.30	62.5	69.4	50.9	58.6	647.3
Apr	4.19	706,461.70	75.1	81.1	68.4	78.9	720.0
May	6.28	781,948.20	77.4	76.1	68.6	62.3	642.0
Jun	4.28	733,323.10	82.6	83.1	76.6	54.1	444.0
Jul	3.92	724,836.80	80.8	87.7	76.5	56.4	454.8
Aug	2.06	767,085.70	84.2	82.0	77.8	44.1	394.5
Sep	1.58	625,460.50	82.0	79.9	74.8	42.1	396.0
Oct	2.88	546,972.00	77.3	79.4	69.9	39.3	363.0
Nov	0.88	295,750.70	72.2	81.7	65.9	64.8	581.8
Dec	0.22	278,694.70	69.3	85.9	64.5	71.0	628.3
Grand Total	28.19	6,950,873.30	74.8	80.8	67.9	60.4	6619.5

Appendix C. US Army Aberdeen Test Center Hydrogen Embrittlement Report

US Army Aberdeen Test Center
Warfighter Directorate
Applied Science Test Division
Materials & Measurements Test Branch
400 Colleran Road, Building 400, Aberdeen Proving Ground, MD 21005-5059

Conducted For:

John Kelly
US Army Research Laboratory
RDRL-WM-CM
john.v.kelley8.civ@mail.mil

MMTB Test Report # : 2013-MM-032

Report Title: Hydrogen Embrittlement of Unknown Coatings

Any reproductions or excerpts from this report must reference the entire report or the report number. The results relate only to the specific samples/test item/test scenario identified within this report.

Report Prepared By:

William H. Taylor
410-278-4461
wglliam.h.taylor11.civ@mail.mil

Jeffrey J. Hosto, Branch Chief
410-278-5306
jeffrey.j.hosto.civ@mail.mil

Approved: ________________________ Date: 17 Jun 13

ADRIENNE H HUGHES
Chief, Applied Science Test Division

1.0 Objective

The objective of this test is to determine the hydrogen embrittlement characteristics of various coatings. The hydrogen embrittlement effect the coatings have on materials is determined using ASTM F519 - 12a Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments.

2.0 Criterion

The coatings shall not cause hydrogen embrittlement using type 1d specimens.

3.0 Test Procedures

a. Inspected all coupons to determine if they conformed to the requirements of the lot acceptance criteria for Type 1d notched specimens, as stated in Table 1 of ASTM F519.

b. A sampling of 10 un-plated coupons were used to determine the notch fracture strength (NFS) of the lot and if the coupons are of such similarity to produce consistent results. The coupons were compressed in a fixture or tensile/compression machine to determine the force required to break each coupon, and the distance each coupon was compressed at the time of failure. These values were recorded.

c. Determined if the change in diameter at fracture for each coupon was within 0.008" of the average.

d. The sensitivity to hydrogen embrittlement was demonstrated for each lot by exposing six trial specimens to two different embrittling environments after manufacture.

(1) Three specimens were electroplated under the highly embrittling conditions produced in a cadmium cyanide bath by Treatment A (Table 2), ASTM F519.

(2) Three specimens were electroplated under the less embrittling conditions produced in a cadmium cyanide bath by Treatment B (Table 2), ASTM F519.

(3) The six coupons were loaded to the sustained load of 75% of the NFS. The lot was declared of suitable sensitivity if all three specimens plated by Treatment A fracture within 24 hours and none of the three specimens plated by Treatment B fracture within 200 hours.

e. Four test specimens were used to determine the hydrogen embrittling effect of the coatings.

f. Determined the service environment required for testing as this dictates the percent of NFS to be used for all test coupons. A determination was made if the test coupons were to be plated or tested bare.

g. Loaded the test specimens using the self-loading bolt device. The nut and bolt was cadmium plated to avoid any galvanic reaction with the test specimen. Applied the percentage of NFS loads as stated for the appropriate service environment. Stress levels could have been related to the percentage change in diameter required to fracture the specimens as previously determined during lot acceptance testing.

h. The coating was considered non-embrittling if none of the test specimens failed within 150 hours after being loaded to the prescribed percentage of NFS. Recorded the time to failure if less than 150 hours. The test was discontinued after 150 hours.

i. If one coupon fails before the 150 hour period, the remaining may be step loaded in accordance with the ASTM to determine if the coating is embrittling or non-embrittling.

j. If two or more failed within the sustained load exposure time, the coating process shall be considered embrittling.

3.1 Test Findings

a. All coupons met the dimensional and physical requirements of table 1 in the ASTM.

b. Sixteen un-plated coupons were compressed in an Instron 3382 tensile/ compression machine (Calibrated on 13 Mar 2013 and due re-calibration on 2013 Mar 14). Only ten were required, but 16 were available and produced better averaged results. The results of testing the 16 baseline coupons are below in Table 1.1-1.

Table 1.1-1. Results of loading 16 baseline coupons

	Compressive extension at Maximum Compressive load (in)	Maximum Compressive load (lbf)(ref)	Difference from average, must be < .008"
1	0.0747	501.726	0.0018
2	0.0759	498.546	0.0006
3	0.0779	475.311	-0.0014
4	0.0763	472.131	0.0002
5	0.0752	493.620	0.0013
6	0.0756	488.175	0.0009
7	0.0760	498.647	0.0005
8	0.0790	451.125	-0.0025
9	0.0840	508.422	-0.0075
10	0.0808	487.511	-0.0043
11	0.0784	467.902	-0.0019
12	0.0693	426.295	0.0072
13	0.0698	453.488	0.0067
14	0.0769	456.276	-0.0004
15	0.0779	460.694	-0.0014
16	0.0761	485.197	0.0004
avg	0.0765		0.0000

c. The averaged value for the NFS was determined to be 0.0765".

d. All coupons met the requirement that the change in diameter at fracture load for each coupon must be within 0.008" of the average.

e. The load for the qualification coupons was 75% of the NFS or 0.0497".

f. All qualification and test coupons were loaded by means of a fine threaded bolt and nut using a calibrated dial indicator to change the diameter of each coupon by the amount required.

g. Three treatment B specimens were plated, baked and were then loaded to 75% of the NFS value. These specimens did not break within 200 hours.

h. Three treatment A specimens were plated, not baked and were then loaded to 75% of the NFS value. These specimens did break within 200 hours.

i. The test coupons were taken to Army Research Lab (ARL) to have the coating applied. Four coupons for each coating were tested. The coupons returned in plastic bags marked with the following letters: P, B, Z, A, W, E, X, S, NOR

j. As described in the ASTM, the coatings were determined to be passive coatings and required a sustained load of 65% or 0.0573" of NFS for 150 hours. This load was applied to all of the test coupons exactly as paragraph f.

k. All of the test coupons were placed into a desiccator cabinet to prevent any reaction with humidity in the air.

l. After the required 150 hours all coupons were inspected under 25X magnification. There were no indications of cracks on any test coupon.

4.0 Technical Analysis

All of the applied coatings are considered non-embrittling.

List of Symbols, Abbreviations, and Symbols

APG	Aberdeen Proving Ground
ARL	US Army Research Laboratory
ATC	US Army Aberdeen Test Center
CARC	chemical agent–resistant coating
CCAFS	Cape Canaveral Air Force Station
CR(VI)	hexavalent chromium
CRS	cold rolled steel
DFT	dry film thickness
DI	deionized
DOD	Department of Defense
DTM	direct-to-metal
ESTCP	Environmental Security Technology Certification Program
GPM	gallons per minute
HVLP	high volume, low pressure
IAW	in accordance with
LEAD	Letterkenny Army Depot
mpy	mils per year
MRAP	Mine-Resistant Ambush Protected
NaCl	sodium chloride
NCP	NCP Coatings Inc
NIOSH	National Institute for Occupational Safety and Health
OEM	original equipment manufacturer
QPD	qualified product database
RTU	Ready-to-Use
VOC	volatile organic compounds
TACOM	US Army Tank-automotive and Armaments Command

1 (PDF)	DEFENSE TECHNICAL INFORMATION CTR DTIC OCA
2 (PDF)	DIR ARL RDRL CIO L IMAL HRA MAIL & RECORDS MGMT
1 (PDF)	GOVT PRINTG OFC A MALHOTRA
4 (PDF)	ARL RDRL WMM C J KELLEY T CONSIDINE T BRASWELL A FARRELL